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Evaluation of the Flash Desulfurization Process for Coal Cleaning

Institute of Gas Technology, Chicago, IL

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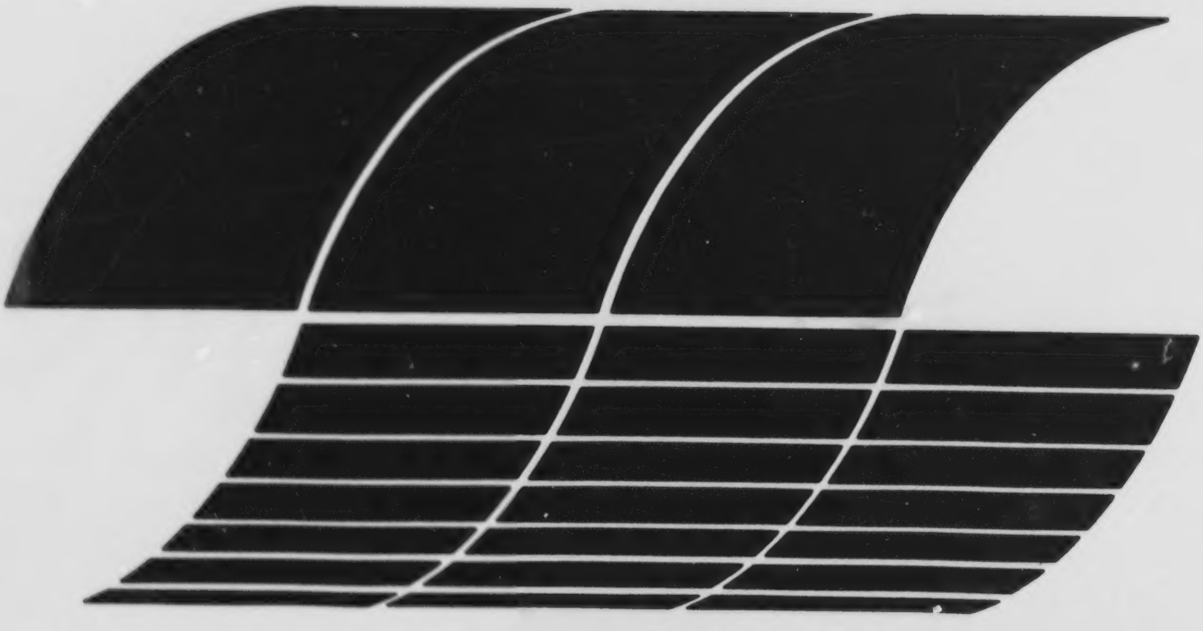
Interagency Energy/Environment R&D Program Report



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16. ABSTRACT

The report gives results of a program to develop (on the laboratory, bench, and pilot scale) operating conditions for key steps in the 'flash' process for desulfurizing coal by chemical and thermal treatment. Laboratory and bench scale data on high-sulfur eastern U.S. coals prove that the process can reduce sulfur to the point that the resulting solid fossil fuel can be directly consumed in compliance with current regulations for SOx emissions. Because of operating and technical difficulties, pilot scale test data are inconclusive. A preliminary analysis of a conceptual process indicates that the treated fuel would cost $1.50 to 1.75/million Btu (in 1977 dollars on a utility financing basis) if the initial coal cost is $1.00/million Btu. Four eastern U.S. coals, from abundant seams, were treated under various reducing-gas atmospheres at elevated temperatures. Sufficient sulfur was removed from all coals tested at ambient pressure and at temperatures of 1500 F and residence times of 60 minutes. These data were obtained in laboratory, fixed-bed, continuous weighing reactors and bench-scale fluidized-bed systems. As conceived, the process incorporated a 'sulfur-getter'--a material (e.g., lime) that has a greater chemical affinity for the sulfur than the coal has. Use of a sulfur-getter is required to reduce the H2S concentration in the gas. Data indicate that the concept is sound.

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EXECUTIVE SUMMARY

The purpose of this program is to develop, on the laboratory-, bench-, and pilot-unit scale, the operating conditions for the key steps in the IGT process to desulfurize coal by chemical and thermal treatment.

Laboratory- and bench-scale data on high-sulfur, Eastern U.S. coals prove that the process is capable of sufficient sulfur reduction that the resulting solid fossil fuel could be directly consumed in compliance with current environmental regulations for sulfur oxide emissions. Because of operating and technical difficulties, the data from the pilot-unit scale testing are inconclusive. A preliminary economic analysis of a conceptual process indicates that the treated fuel would have a price of $1.50 to $1.75/million Btu (in 1977 dollars on a utility financing basis) if the initial coal costs $1.00/million Btu.

An oxidative pretreatment of the coal – air fluidization of the coal at 750°F for 30 minutes with 1 SCF O_2 consumed per pound of dry coal – is an integral part of the system. This pretreatment not only prevents caking in the subsequent fluidized-bed hydrodesulfurization reaction, it also renders the sulfur in the coal more amenable to removal.

Four Eastern U.S. coals, from abundant coal seams, were treated under various reducing-gas atmospheres at elevated temperatures. Sufficient sulfur removal was achieved from all coals tested at ambient pressure and at temperatures of 1500°F and residence times of 60 minutes. These data were obtained in laboratory, fixed bed, continuous weighing reactors and bench-scale, fluidized-bed systems.

As initially conceived, the process incorporated a "sulfur-getter" – a material that has a greater chemical affinity for the sulfur than the coal has. Lime is an example of such a sulfur-getter. Parallel tests in the laboratory- and bench-scale equipment indicated that the addition of lime to the coal during hydrotreatment had little apparent advantage for sulfur removal and significant disadvantages in the recovery of treated material and the regeneration of the lime. Consequently, the use of the sulfur-getter in the process was deleted.

Scale-up problems, however, later proved that the sulfur-getter concept was required in the system. Tests with larger-scale, continuous feeding fluidized-bed equipment did not result in satisfactory sulfur removal at the operating conditions determined from the smaller-scale tests. The necessary ratio of hydrogen-to-coal in the larger units was much lower than in the smaller units; consequently, the hydrogen sulfide concentration in the fluidizing gas was increased, inhibiting desulfurization. The use of the sulfur-getter is therefore required to reduce the hydrogen sulfide concentration in the gas. This discovery was made late in the program and testing of this factor in the large-scale equipment could not be accomplished within the time and budget constraints. Thermodynamic, laboratory, and bench-scale data indicate that the concept is sound.

A conceptual process design was developed, based on the process without the getter addition. Heat and material balances were made. Significant by-product values in power, intermediate-Btu gas, BTX, and sulfuric acid reduced the price of the desulfurized solid fossil-fuel product to about $1.29/million Btu. A process was conceptualized including iron oxide as a sulfur-getter (based on the contractor's Steam-Iron Process technology) and, using very conservative processing assumptions in the absence of hard data, the cost of the treated fuel product was approximated at $1.75/million Btu.

A brief outline for the direction of future work on the process is indicated, including the construction and operation of a continuous, integrated Process Development Unit for proof of the technical viability of the process.

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CONVERSION FACTORS

Non SI Units	Operation	SI Unit
atmosphere	x 101325	N/m^2
Btu	x 1055.87	J
Btu/lb	x 2327.794888	J/kg
cal	x 4.19002	J
^oC	+ 273.15	K
^oF	$(5/9)(T_F + 459.67)$	K
$^oF/min$	x 0.0092593	K/s
foot	x 0.3048	m
gpm (gallons per minute)	x 0.0000630902	m^3/s
inch	x 0.0254	m
in H_2O	x 249.082	N/m^2
in H_2O/ft	x 2988.98	N/m^3
pound	x 0.45359237	kg
psi(a)	x 6894.7572	N/m^2
SCF/hr	x 0.000007865790722	m^3/s

Mesh An Empirical Measure of Particle Size:

U.S. Mesh	Opening Size mm
10	2.00
12	1.68
14	1.41
20	0.84
30	0.59
40	0.42
60	0.25
80	0.177
100	0.149

OBJECTIVE

The objective of the program is to determine experimentally, on laboratory-, bench-, and pilot-unit scales, the operating conditions for the key steps in the IGT process to desulfurize coal by thermal and chemical means. The current (early 1978) Federal EPA New Source Performance Standard (NSPS) for solid fossil-fuel combustion has largely been observed by switching to low-sulfur fuels. Achieving the goals of this program will increase the supply of low-sulfur solid fuels.

INTRODUCTION AND BACKGROUND

This document is the final report under EPA Contract No. 68-02-2126, encompassing work on the development of the Flash Desulfurization Process over the period from November 1975 through October 1977.

Researchers at the Institute of Gas Technology (IGT) have conceived a process for the removal of sulfur from coal by a combination of thermal and chemical means: A U.S. Patent has been granted on this process and assigned to IGT. The objective of the work described in this report is to develop the key steps of that process.

The process incorporates low-pressure (nearly ambient) treatment of the coal in a reducing atmosphere at temperatures sufficient to liberate the sulfur in the coal as H_2S. However, the sulfur in the coal is not a distinct chemical species, but exists in many forms that react with hydrogen at varying temperatures. The program is designed to evaluate the removal of total sulfur (organic as well as pyritic) from the coal as functions of operating temperature and reaction time and to determine the severity of treatment required for the manufacture of an environmentally satisfactory, solid fuel product.

The initial process concept included adding a "sulfur-acceptor" ("sulfur-getter") to the coal. The thermodynamic equilibrium concentration of H_2S over coal-ash is not high and the sulfur-getter is defined here as a material that has a greater affinity for the sulfur than the coal has. One example of such a sulfur-getter is lime: Hydrogen sulfide has a lower equilibrium partial pressure over lime than it has over coal. Therefore, the reducing gas will react with the coal-sulfur, forming H_2S. The H_2S, however, will react almost instantaneously with the lime. In the overall system, therefore, sulfur is transferred from the coal to the lime with an H_2S intermediate.

The work reported here is a continuation of a program reported earlier under Contract No. 68-02-1366 entitled, "Pilot Plant Study of Conversion of Coal to Low-Sulfur Fuel." The summary of that report is presented below as background for this document:

> "The purpose of this program is to develop, on a bench and pilot scale, the operating conditions for the key step in the IGT process to desulfurize coal by thermal and chemical treatment. This process, to date, uses the "sulfur-getter" concept. A sulfur-getter is defined as a material that has a greater chemical affinity for sulfur than the coal has. Lime has been selected as the sulfur-getter for this program.
>
> "The program reported here was divided into two phases. In Phase I, the problem was directly attacked on a pilot-unit scale. The results of this work indicated that the program should be redirected (Phase II) to smaller-scale test apparatus so that more basic data could be obtained for eventual scale-up to pilot scale.

"In the initial project phase, a coal-lime mixture was experimentally treated at atmospheric pressure with a reducing gas in a heated, fluidized-bed reactor. This reactor could treat up to 200 lb/hr of mixture to temperatures of 1200°F. The coal used in the initial tests was from the Illinois No. 6 seam and contained about 3% sulfur.

"Work in the initial program phase resulted in the discovery that less sulfur was removed than expected at these conditions. Two factors were believed responsible:

1. The coal heat-up rate in the fluidized bed was nearly instantaneous, which appears to cause organic sulfur fixation.

2. The coal showed signs of weathering; therefore, the total sulfur content was not readily available for hydrogen treatment.

"At this point, the program was redirected (Phase II) to the operation of smaller-scale test units that featured controlled heat-up rates. The smaller-scale units also permitted an increased number of tests over a broader range of conditions, with savings in time and manpower. Coal samples from several mines throughout the country were obtained for tests in this equipment.

"A coal-lime mixture was treated with hydrogen, in batch-type reactors, to temperatures of 1500°F. Heat-up rates, terminal temperature, residence time, and particle size were the variables tested.

"Preliminary tests eliminated the Western coals (i.e., subbituminous and lignite) because the sulfur content of the raw coal was low and readily amenable to treatment. Also, the preliminary tests indicated that the coals from the Midwestern and Eastern United States required pretreatment to prevent caking during hydrotreating. This is accomplished by heating the fluidized coal at atmospheric pressure to 750°F in the presence of oxygen.

"On the basis of the preliminary tests with several coals and the relative abundance of the types of coal, a coal from the Western Kentucky No. 9 seam was chosen for complete characterization. This coal is from the Illinois basin and contains over 3% sulfur. Tests were run covering a wide range of the parameters listed. These tests prove that acceptable sulfur levels were attained at treatment temperatures of 1500°F. The higher temperatures result in significant tar removal and some gasification of the coal. These effects necessitate further research into quantity, chemical makeup, and handling of gas and liquid streams.

"The testing resulted in the discovery that treatment with lime does not capture all the sulfur that is released from the coal. A more thorough examination of the effectiveness and benefits of lime is required in future work.

"A conceptual process design, based on laboratory- and bench-scale data, is presented. That process will produce a solid fuel that can be burned directly in conformance with Federal EPA New Source Performance Standard (NSPS)."

At the end of the previous contract, therefore, a conceptual process had been identified but could not be defined because of an insufficient data base. The section entitled "Future Work" included:

> "Necessity for Lime
>
> "One of the primary objectives to consider is the necessity of using lime. Some data indicate that the use of lime is not imperative; the process could be simplified if the lime were eliminated. Problems have been experienced with coal-lime separations, loss of carbon values to the lime, and capture of lime by the coal. These problems were not unexpected in the original plan for the process, but the effect is more pronounced than desirable.
>
> "Elimination of the lime would reduce the complexity of the process. The reactors could be made smaller, and gas usage would decrease because less material would be handled. Larger off-gas treatment facilities would be necessary to handle the increased sulfur in the off-gas. Studies must be made to determine which operating approach is economically and operationally superior.
>
> "Other Coals
>
> "Tests using other, typical sulfur-bearing coals should be made in the thermobalance and batch reactors. The results will be compared with results already obtained. The relative value of heating rates and holding times will be evaluated.
>
> "Pilot Unit
>
> "Tests should be made on the 10-inch unit used previously. The larger-scale operation and increased material generated are necessary for determining details to complete the process flow sheet. In particular, the determination and distribution of sulfur types in the off-gas during continuous operation are needed, so that treatment facilities can be designed.
>
> "If a heating rate must be imposed on the particle (other than the rapid heat-up in the fluidized-bed arrangement), the 10-inch unit will require modification. Further work is needed in the thermobalance and batch reactors to determine the optimum heat-up rate.
>
> "Overall Concept Design
>
> "When test work is completed, data would be used to generate an overall conceptual design for the process. This would include energy and material balances, economic studies, and all of the treatment steps to produce a low-sulfur fuel from coal."

The present work has been directed at proving these points.

The organization of this report is as follows: Detailed discussion of materials, equipment, and operating procedures are omitted; these subjects are fully described in the above-mentioned report on the earlier program phases. Where new coals, equipment, or techniques were employed, these are described in the section of the report pertaining to their use.

The first section of the report, Coal Preparation, discusses experimental efforts in the plant section upstream of the hydrodesulfurization reactor. Work was done on coal crushing and coal pretreatment.

Discussions of the hydrodesulfurization reaction are presented in the order of increasing equipment size — laboratory, bench-scale, and 10-inch continuous feeding systems. A short discussion is presented on coal nitrogen reduction during hydrodesulfurization, and combustion methods for utilization of the treated coal are identified.

The economic studies include the development of a conceptual process. Also, the equipment requirements are described for a continuous Process Development Unit (PDU) for proof of the overall process concept on an integrated basis.

Finally, the next steps in the development of the system are outlined.

COAL PREPARATION

CRUSHING

A quantity of Illinois No. 6 coal was obtained. Analyses for the run-of-mine coal are given in Table 1. It is much higher in ash and sulfur content and lower in fixed carbon than earlier samples of washed coal.

This coal was used in crushing tests by the T.J. Gundlach Machine Co. The purpose of the tests was to determine conditions to minimize fines in crushing and preparation. These data will be used for design work. Gundlach's crusher has the advantage of reduced fines production over other types of crushers.

Three tests were used with one pass of coal through the crusher. Table 2 lists the screen analysis for feed and the material produced from each test; the feed material was initially screened at 10 mesh to remove the fines material from the feed. The three tests were made at crusher speeds of 200, 300, and 400 rpm. The merits of each can be seen in the screen analysis. Lower speeds produced fewer fines, but increased the amount of oversize material to be recycled for recrushing. Increasing the speed reduces the amount recycled, but increases the fines. Some of the material was crushed to 100% -8 mesh for use in pretreatment and desulfurization tests.

Other work at the contractor's site indicated that the top size required for satisfactory operation of an 8-foot-diameter pretreater was about 14 mesh. Screening of mid-continent coals at 12 mesh resulted in a sufficiently low quantity of +14 mesh coal that pretreatment was acceptable in that pilot plant unit.

Analysis of the crushing tests, assuming a closed-loop crushing around a 12 mesh screen, indicates that the crushed and screened product should contain less than 12% —100 mesh material. This feed should be satisfactory for the operation of the pretreater and hydrodesulfurization reactor with minimum elutriation of fines.

PRETREATMENT

Many U.S. coals require a step called pretreatment before they can be satisfactorily processed in fluidized-bed reactors at elevated temperatures. Tests with the reaction system proposed for this program indicated that pretreatment will also be required in the coal desulfurization process.

Laboratory- and bench-scale hydrodesulfurization tests prove that pretreatment not only prevents caking within the reactor, but it also increases the sulfur removal in the subsequent hydrotreating step. Figure 1 represents two series of thermobalance tests made with Western Kentucky No. 9 coal. One test series was made with crushed and screened coal; the other test used crushed, screened, and pretreated coal as feed for hydrodesulfurization. The results show that the 70% sulfur removal (based on the initial feed), as achieved with untreated coal feed, was increased to 95% by using a pretreated

TABLE 1. ILLINOIS NO. 6 COAL ANALYSIS (HILLSBORO MINE)

Proximate Analysis, wt %	
Moisture	12.02
Ash	22.83
Volatile Matter	30.18
Fixed Carbon	34.97

Ultimate Analysis, wt % (dry)	
Ash	25.95
Carbon	57.07
Hydrogen	4.01
Sulfur	5.06
Nitrogen	0.98
Oxygen	6.82

Heating Value, Btu/lb	10,198

TABLE 2. SCREEN ANALYSIS FOR CRUSHING TESTS

% Retained in Indicated Sieve	Feed	200 rpm	300 rpm	400 rpm
2 in.	0	0	0	0
1 in.	8.6	0	0	0
3/4 in.	10.1			
1/2 in.	15.9	2.9	0.6	0
1/4 in.	29.8	13.2	5.1	2.6
6 mesh	13.7	23.7	14.1	7.2
8 mesh	7.7	13.8	10.6	6.7
12 mesh	7.0	13.0	12.8	9.3
20 mesh	3.9	16.9	24.4	22.9
50 mesh	1.0	9.1	19.6	28.1
70 mesh	0.4	2.1	4.0	6.9
100 mesh	0.3	1.4	2.6	4.8
Pan	1.6	3.9	6.2	11.5

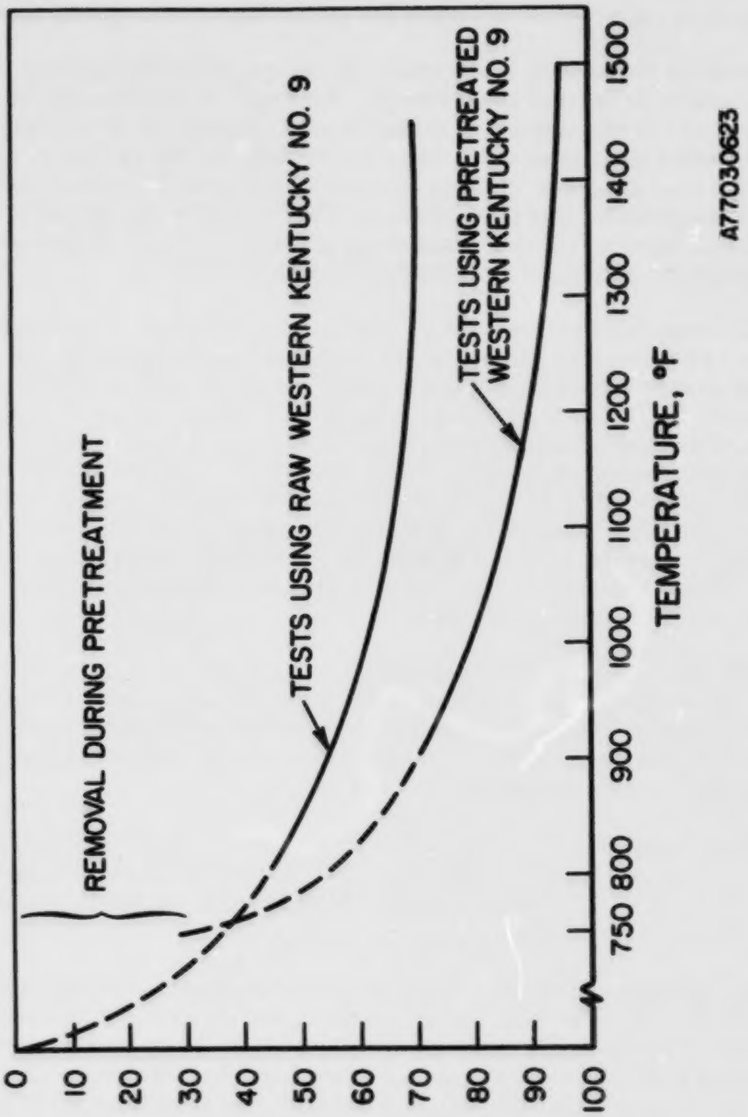

Figure 1. Sulfur removal indicating effect of coal pretreatment.

feed. Note that this increased removal was not due to sulfur removed in pretreatment: Sulfur removed initially during pretreatment, primarily pyritic sulfur, is also readily attacked in low-temperature (800° to 1000°F) hydrotreatment; the improved sulfur removal after pretreatment is obtained by more complete removal of organic-type sulfur. The more complete removal may be caused by the increase in coal pore size during pretreatment, resulting in better contact of the hydrogen with the coal sulfur and larger passages for removal of the resultant hydrogen sulfide. Also, oxidative treatment of sulfur compounds is expected to activate the sulfur for further reactions.

Pretreatment is required in many coal conversion processes and has been the subject of investigation at several laboratories. Even so, the mechanism of pretreatment is little understood and is the subject of disagreement among coal investigators. The discussion here is based upon a) specific tests conducted for this program, b) tests conducted for a parallel program utilizing the same coal, and c) previous data obtained at IGT and other laboratories on this phenomenon. The data from the parallel program provide a substantial input to this understanding, and the sponsors of that program have consented to release the results of that study into this project.

Many coals from the Eastern United States are considered to be "agglomerating." Historically, this agglomerating tendency was described as a "softening" of the coal particle as it was heated through its "plastic range" – about 600° to 900°F. The softened coal particles would adhere to each other, causing an increase in the average particle size, characterized as agglomerating or caking. These coals, when examined by time-lapse or slow-motion microphotography of heated particles, would actually turn fluid in the plastic range and resolidify at higher temperatures. The explanation presented for this effect was a softening of the coal, actually dissolving into its low-melting constituents, followed by polymerization of the melt to a different form of coal, characterized as coke or char. These studies, at other laboratories, have provided a graphic illustration of the phenomenon that must be overcome.

The historic approach to pretreatment is light oxidation of the coal particles with air at temperatures in the range of 700° to 800°F. Other approaches have also been considered, but the air-pretreatment appears well suited for incorporation into the proposed flowsheet. The purpose of the pretreatment study here was to better define the conditions necessary for this operation.

The only real measure of satisfactory pretreatment is non-agglomeration of the coal in the downstream processing equipment. This effect is not readily measured in the analytical laboratory, so other tests have been devised for laboratory evaluation of the caking characteristics of the coal.

- Free-swelling index: This laboratory test measures the swelling of coal mass while being heated. The test was developed primarily to indicate effects that might occur with different coals in a coke oven. In general, a coal with a high free-swelling index will be caking, and some investigators claim that reduction of the free-swelling index to the range of 1.0 to 2.0 is an indication of satisfactory pretreatment.

- Reflectant borders: When air-pretreated coal is examined microscopically, the edges of the particles show a greater light-reflectance than the interior of the coal. It has been hypothesized that the edges of the particles either may have a high ash content or may be coked by the action of the air, forming a skin. This skin is assumed to retain the fluids generated as the coal is heated, and to keep the surface of the particle from becoming adherent.

- Boat test: The coal is heated under inert atmosphere with a programmed time/temperature profile while contained in a boat made of stainless steel screen. The resultant material may a) fuse into a single cake for severely agglomerating coals, b) sinter into a porous block for mildly caking coals, or c) be free flowing after the test. Intermediate degrees of caking can be noted, but the test is subjective for all degrees of caking worse than a free-flowing material.

Based upon the results of this recent work, the boat test is considered to be the best measure of satisfactory pretreatment. Work in other studies has succeeded in reducing the free-swelling index to low values, but the material still agglomerates in the boat test. Also, in this work it has been found that some coals may be satisfactorily pretreated, even if no reflectant borders are generated. Although the boat test is subjective, yielding results that cannot be quantified numerically, and only indicates a "go or no-go" situation, it has been found to give results that are safest in assuring satisfactory operation in downstream equipment.

In addition to the laboratory tests, a visual examination of a pretreater effluent provides a qualitative estimate of satisfactory operation of the unit. The raw coal is angular, but, during pretreatment, the coal swells into rounded particles. The occurrence of this effect is not sufficient evidence of pretreatment, but provides the operator with a quick, qualitative estimate of successful operation.

An extensive series of tests in both the modified batch reactor and the 10-inch-diameter pilot unit has been conducted on the Western Kentucky No. 9 coal to evaluate pretreatment conditions. The operation of these experimental units and the techniques of analysis of the feed and product streams have been detailed in the Final Report under EPA Contract No. 68-02-1366 (the previous contract on this coal desulfurization project). At the conclusion of the program under Contract No. 68-02-1366, a series of batch reactor tests had been run under a wide variety of temperatures, oxygen feed rates, and residence times without achieving satisfactory pretreatment. The results that follow are based upon the output of a parallel program that continued during the interim between Contracts No. 68-02-1366 and 68-02-2126. The specific details of the tests are not presented because they are proprietary to that program's sponsor; however, the sponsor has consented to release the results of those tests to this program.

Photomicrographic examination of the residues from the initial, unsatisfactory pretreatment tests indicated that some particles were excessively oxidized while others were essentially untreated, resulting in a mixture that would still sinter in the boat test. Consequently, mixing in the fluidized-bed reactor was found to be an important parameter. The initial tests had been run at linear gas velocities well above the minimum fluidization velocity, but the velocity was not sufficient to ensure full mixing within the bed. Subsequent tests indicated that a velocity of at least 0.75 ft/s, measured at the bed conditions, was required to achieve satisfactory mixing and pretreatment of the −20+80 mesh coal feed. This minimum velocity was probably a function of the mechanical characteristics of the pretreatment vessel, and experience has shown that the mixing velocity might be reduced in larger equipment. However, this velocity is one of the parameters that control the size of the full-scale equipment; higher velocities are desirable to minimize equipment size (but increase fines elutriation). The primary difficulty encountered with the specified mixing velocity is the requirement of additional fluidizing gas, in addition to the reaction air required, in some smaller-scale tests.

A series of tests was next made in the batch reactor with adequate mixing and different quantities of reactant air at a temperature of 750°F. In all cases, boat tests proved that the pretreatment was adequate. Photomicrographic examination showed the thickness of the reflectant coal border varied directly with the quantity of air used. Satisfactory pretreatment was even accomplished with this coal in a nitrogen atmosphere, indicating that the formation of a protective skin on the coal particles is not a neccesary condition for satisfactory pretreatment of this particular coal. In general, it was found that satisfactory pretreatment resulted if the volatile matter content of the coal was reduced below a specific level by any combination of time, temperature, or oxygen consumption that would achieve this devolatilization.

Although the volatile matter content of the pretreated coal appeared to be the controlling parameter for this coal, the mechanism for achieving this devolatilization must be considered. After examination of energy balances, material consumption, and energy requirements of the overall desulfurization system, the specifications for the pretreatment system were written. These specifications included the following:

- Operating temperature – 750°F
- Minimum residence time – 30 min
- Oxygen feed – 1 SCF O_2/lb dry coal
- Fluidization velocity – 1 ft/s measured at operating conditions.

Analyses for raw coal and coal pretreated at these conditions are given in Table 3. A flow sheet based upon the results of operation of the 10-inch unit for the Western Kentucky No. 9 coal is shown in Figure 2.

With the operating conditions described, the pretreater off-gas contains significant heating value in the form of reducing gases, tars, and coal fines. The fines would largely be returned to the bed by internal cyclones in a full-scale unit. Cooling of the off-gas for desulfurization and separate recovery of tars and fines should not be done, because of the significant sensible heat that would be lost. The gas should be burned directly (as in a CO-boiler) for recovery of energy for input elsewhere in the process, followed by desulfurization of the stack gas.

The pretreatment reaction results in the partial removal of sulfur from the coal. For the Western Kentucky No. 9 coal, approximately 28% of the sulfur is removed. The sulfur appears primarily as sulfur dioxide with smaller quantities of carbonyl sulfide and traces of other organic compounds. Hydrogen sulfide does not appear to be a significant product of this pretreatment reaction.

The operating conditions for the above work were used in several runs in the 10-inch-diameter continuous units. A confirmation of the batch reactor tests and production of a quantity of pretreated char for subsequent hydrodesulfurization tests resulted.

A similar study was done with Illinois No. 6 coal. The batch reactor was used with various amounts of oxygen usage and treatment times. On the basis of the tests, an air consumption of 1 SCF O_2/lb of coal with a 30-minute residence at 750°F was found to be sufficient for adequate pretreatment.

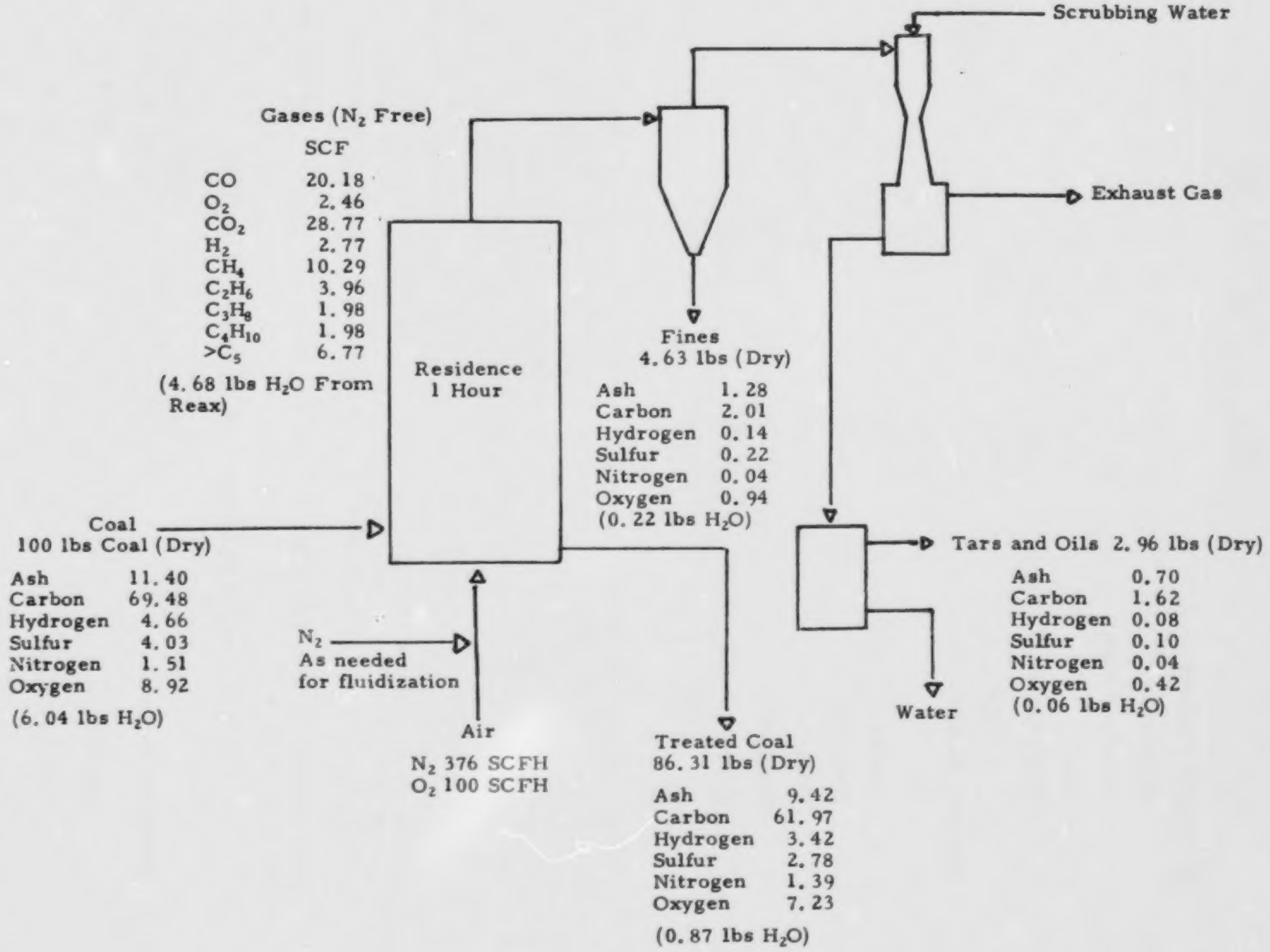
Scrubbing Water
Exhaust Gas
Gases (N_2 Free)
SCF
CO 20.18
O_2 2.46
CO_2 28.77
H_2 2.77
CH_4 10.29
C_2H_6 3.96
C_3H_8 1.98
C_4H_{10} 1.98
$>C_5$ 6.77
(4.68 lbs H_2O From Reax)
Residence 1 Hour
Fines
4.63 lbs (Dry)
Ash 1.28
Carbon 2.01
Hydrogen 0.14
Sulfur 0.22
Nitrogen 0.04
Oxygen 0.94
(0.22 lbs H_2O)
Coal
100 lbs Coal (Dry)
Ash 11.40
Carbon 69.48
Hydrogen 4.66
Sulfur 4.03
Nitrogen 1.51
Oxygen 8.92
(6.04 lbs H_2O)
N_2
As needed for fluidization
Air
N_2 376 SCFH
O_2 100 SCFH
Treated Coal
86.31 lbs (Dry)
Ash 9.42
Carbon 61.97
Hydrogen 3.42
Sulfur 2.78
Nitrogen 1.39
Oxygen 7.23
(0.87 lbs H_2O)
Water
Tars and Oils 2.96 lbs (Dry)
Ash 0.70
Carbon 1.62
Hydrogen 0.08
Sulfur 0.10
Nitrogen 0.04
Oxygen 0.42
(0.06 lbs H_2O)

Figure 2. Pretreater flowsheet (basis: 100 lb dry Western Kentucky No. 9 coal).

TABLE 3. TYPICAL ANALYSIS OF RAW AND PRETREATED COAL

	Coal	Pretreated Coal
	wt %	
Proximate Analysis		
Moisture	5.8	0.8
Volatile Matter	36.3	27.7
Ash	10.6	11.2
Fixed Carbon	41.3	60.3
Total	100.0	100.0
Ultimate Analysis		
Ash	11.24	11.25
Carbon	70.00	71.40
Hydrogen	4.54	4.06
Sulfur	3.74	3.16
Nitrogen	1.53	1.64
Oxygen	8.95	8.49
Total	100.00	100.00

The 10-inch unit was operated to pretreat the Illinois No. 6 coal. The conditions determined from the batch reactor runs were used as operating guidelines. In this test, the feedstock was Illinois No. 6 coal that had been crushed to a size consist of −14 mesh by 0. The feed rate was approximately 50 lb/hr, yielding a residence time in this unit of 40 minutes. The air flow rate was adjusted to provide an oxygen addition of 1 SCF O_2/lb of coal fed. Additional nitrogen was added to the gas flow to ensure sufficient fluidization. The operating temperature during the steady-state portion of the run was 750°F. These operating conditions represent approximately the minimum severity for effective pretreatment, as determined from the batch reactor runs reported above. The pretreatment of the coal was satisfactory during this run, as evidenced by the free-flowing characteristics of the product in the empirical boat test for agglomeration.

The proximate analysis for the feed and the pretreated coal product of this test is presented in Table 4.

TABLE 4. PRETREATMENT OF ILLINOIS NO. 6 COAL

Proximate Analysis	Feedstock Coal, wt %	Pretreated Coal, wt %
Moisture	2.3	0.8
Volatile Matter	34.0	24.6
Ash	8.1	8.2
Fixed Carbon	55.6	66.4

In examining this proximate analysis, the data for this test appear suspect — although the volatile matter content of the pretreated coal has been reduced to the range normally associated with satisfactory pretreatment, the ash content of the pretreated coal is nearly identical to the feedstock. This similarity in ash content would indicate minimum reaction of the oxygen with the coal. Examination of the ultimate analyses of all streams around the pretreater, as presented in Figure 3, indicates other inconsistencies with prior pretreatment tests. For example, the total mass of pretreated coal, fines, and scrubber material is 97.7% of the feedstock, indicating that only 2.3% of the coal is consumed into off-gases and water. Further, the hydrocarbon content of these off-gases is low — only 10 Btu/SCF.

Although the raw data appear suspect, the material balances (both overall and component) presented in Table 5 are excellent. These good material balances add credibility to the data. Therefore, results are presented here with the caveat that, although the data appear good, they do not agree with earlier results, and further pretreatment testing is required.

The development of the material balances requires further explanation. First, because of the equipment configuration, the cyclone underflow (fines) and scrubber discharge cannot be quantitatively separated for the steady-state portion of the run. For these tests, therefore, the total material collected in the cyclone underflow and the scrubber was allocated to the steady-state portion of the run according to the time of operation. The excellent ash balance for the run verifies this calculational technique. The equipment, also, does not permit direct determination of the quantity of water manufactured in the pretreater. Two approaches are possible for estimating this water production: forcing the hydrogen balance or forcing the oxygen balance. Neither of these approaches is entirely satisfactory. The oxygen contents of the solid materials are determined by difference, and errors in any of the analyses will be reflected in the oxygen content reported. However, forcing the hydrogen balance requires the differencing of similar numbers and the high leverage of change in hydrogen weight against amount of water produced. For this material balance, the decision was to force the oxygen balance, primarily because of the leverage factor in forcing the hydrogen balance. One other calculational point in the material balance is that the air addition was calculated without the addition of the excess fluidizing nitrogen — as would be experienced in a larger unit — and the off-gas compositions were back-calculated to account for this effect. In general, however, the total and component material balances for the test are excellent.

The heat balance for the pretreatment test is presented in Table 6. The balance as presented agrees within 0.1%, with slightly greater heat output than input. This balance is calculated on the basis of a larger unit, without the use of the excess fluidizing nitrogen and electric heaters that are employed on the test run with the 10-inch-diameter reactor. These data indicate that no excess heat would be available for steam generation under these conditions — in contrast to earlier small-scale tests, but tend to support the operation of larger scale units as extrapolated to these operating conditions.

An overall evaluation of these data indicate that, on the commercial scale, the recovery of heat content in the coal across the pretreatment section would be excellent. The test apparatus has no system for recovery of fines, but, in a larger unit, the fines would report to the product because of internal cyclones. Therefore, referring to Figure 3, over 90% of the dry coal input would be expected in the pretreated product.

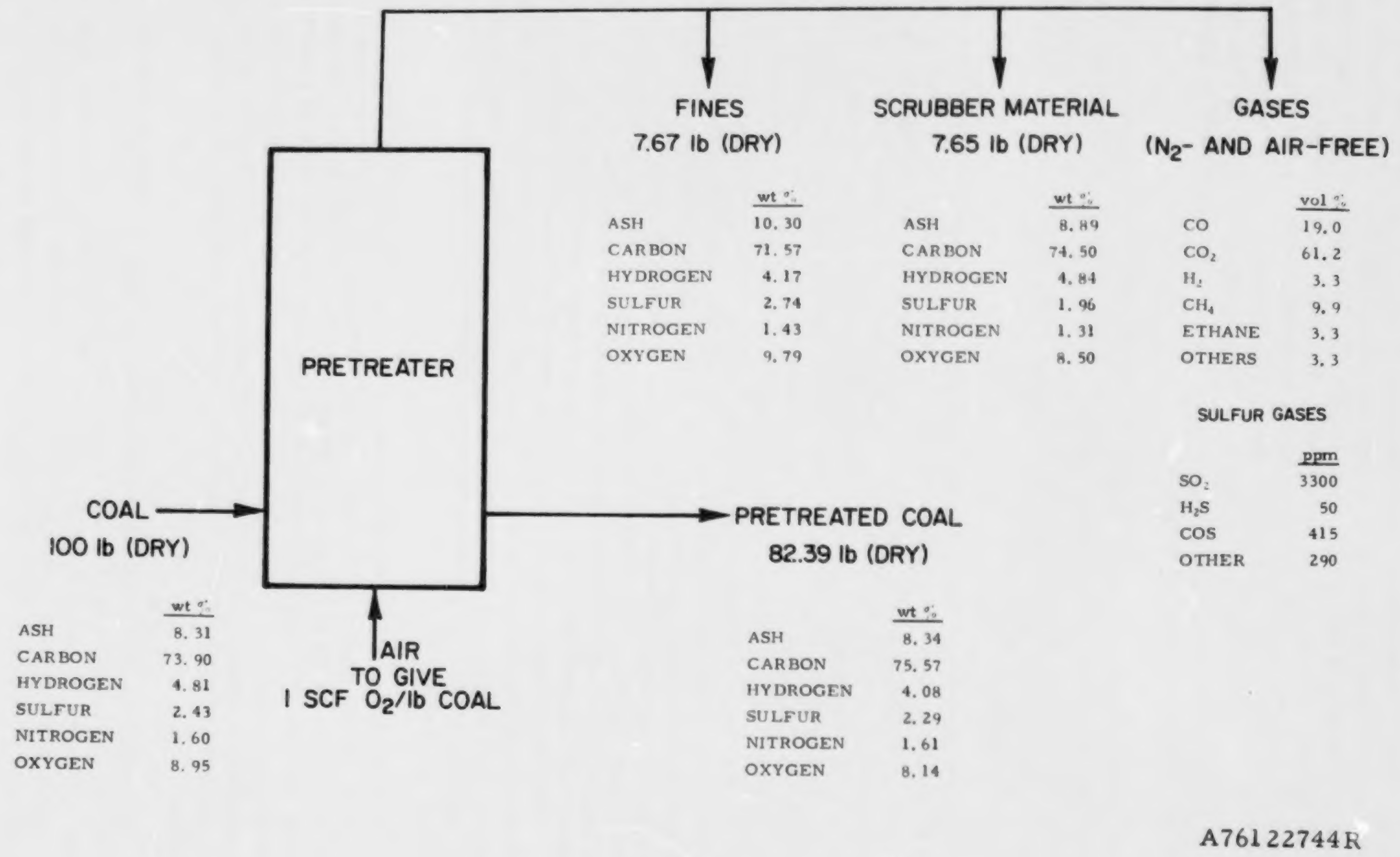
PRETREATER
COAL
100 lb (DRY)
wt %
ASH 8.31
CARBON 73.90
HYDROGEN 4.81
SULFUR 2.43
NITROGEN 1.60
OXYGEN 8.95
AIR
TO GIVE
1 SCF O2/lb COAL
FINES
7.67 lb (DRY)
wt %
ASH 10.30
CARBON 71.57
HYDROGEN 4.17
SULFUR 2.74
NITROGEN 1.43
OXYGEN 9.79
SCRUBBER MATERIAL
7.65 lb (DRY)
wt %
ASH 8.89
CARBON 74.50
HYDROGEN 4.84
SULFUR 1.96
NITROGEN 1.31
OXYGEN 8.50
GASES
(N2- AND AIR-FREE)
vol %
CO 19.0
CO2 61.2
H2 3.3
CH4 9.9
ETHANE 3.3
OTHERS 3.3
SULFUR GASES
ppm
SO2 3300
H2S 50
COS 415
OTHER 290
PRETREATED COAL
82.39 lb (DRY)
wt %
ASH 8.34
CARBON 75.57
HYDROGEN 4.08
SULFUR 2.29
NITROGEN 1.61
OXYGEN 8.14
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Figure 3. Pretreater material balance, Illinois No. 6 coal.

TABLE 5. PRETREATER MATERIAL BALANCE
ILLINOIS NO. 6 COAL

Material In

	Coal (Dry)	Air	Total
Weight, lb	100	38.25	138.25
Ash	8.31		8.31
Carbon	73.90		73.90
Hydrogen	4.81		4.81
Sulfur	2.43		2.43
Nitrogen	1.60	29.34	30.94
Oxygen	8.95	8.91	17.86

Material Out

			Scrubber			
	Pretreated Coal	Fines	Solids	Tars & Oils	Off-Gases	Total
Weight, lb	82.39	7.67	6.41	1.24	40.76	138.47
Ash	6.84	0.79	0.68	0.00	0.00	8.31
Carbon	62.26	5.49	4.63	1.07	0.45	73.90
Hydrogen	3.36	0.32	0.26	0.11	0.98	5.03
Sulfur	1.89	0.21	0.13	0.02	0.18	2.43
Nitrogen	1.33	0.11	0.10	0.00	29.40	30.94
Oxygen	6.71	0.75	0.61	0.04	9.75	17.86

TABLE 6. PRETREATER HEAT BALANCE
(BASIS: 77°F, 100 lb DRY COAL)

HEAT IN

	lb		C_p, Btu/lb-°F	Enthalpy, Btu	Heating Value, Btu/lb	Total Heating Value, Btu	Total, Btu
Coal, Dry	100.0		0.254	0	13,169	1,316,900	1,316,900
Water in Coal	2.3		1.00	0	0	0	0
Air, 476 SCF			0.24	0	0	0	0
Total In				0		1,316,900	1,316,900

HEAT OUT

	lb		C_p, Btu/lb-°F	Enthalpy, Btu	Heating Value, Btu/lb	Total Heating Value, Btu	Total, Btu
Pretreated Coal	82.39		0.254	14,084	13,022	1,072,882	1,086,966
Fines	7.67		0.254	1,311	12,449	95,484	96,795
Scrubber Material							
Tars	1.24		0.446	372	17,188	21,313	21,685
Solids	6.41		0.254	1,096	12,698	81,394	82,490
Gases		SCF			Heating Value, Btu/lb-mol		
N_2	29.40	377.0		8,918	--	--	8,918
O_2	0.83	9.3		226	--	--	226
CO	0.18	2.3		55	121,718	782	837
CO_2	0.91	7.4		228	--	--	228
H_2	0.002	0.4		9	123,090	137	146
CH_4	0.05	1.2		42	383,395	1,281	1,323
C_2H_6	0.03	0.4		23	671,634	748	771
Others	--	0.4		30	--	1,600	1,630
H_2O (from reax)	9.18	--		12,925	--	--	12,925
H_2O (in coal feed)	2.31	--		3,252	--	--	3,252
Total Out				42,571		1,275,621	1,318,192

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In the test unit, the scrubber material contains both extreme fines – which escape the external cyclone – and tars. In the analysis of Table 5, these components were separated and show a relatively high quantity of solids. This high quantity of solids is present because the feedstock was not screened for bottom size. In a commercial unit, the heating value of these solids, in addition to the tars and off-gases, would be recovered in a separate boiler.

In summary of this pretreatment test, the data appear excellent because of the good closure of material and energy balances. However, the low quantities of light hydrocarbons in the off-gases and the high solids recovery around the unit – compared with earlier tests – add an element of doubt to the data. A possible explanation of the difference in these test results from the body of data collected in other programs at the contractor's pilot plant lies in the feed size consist of the feedstock and the method of feeding the 10-inch-diameter PDU reactor. This feed had not been double-screened for removal of fines and the feed was introduced at the bottom of the bed, at the point of air injection. It appears that the air preferentially reacted with the coal fines, because of the high surface area of this size fraction, rather than with the bulk of the coal. In this case, the necessary heat release was available to pretreat the mid-continent coal, but other data, more representative of a commerical reactor configuration, should be used for a design basis.

Batch reactor tests were also made with the two Pittsburgh-seam coals to determine pretreatment conditions. Continuous unit runs were not made with these coals because of supply and success with predicting pretreatment conditions with the small reactor.

Results for the West Virginia-mine coal are presented in Table 7. In each test, the coal was fluidized with nitrogen while being heated to 750°F. Air was then added for the time given at a rate to yield 1 or 2 SCF O_2/lb of coal. As expected, this coal needs a more severe pretreatment than the Western Kentucky No. 9. Conditions of 30 minutes pretreatment and an air consumption of 2 SCF O_2/lb of coal for Run BR-P75-1 were not sufficient to prevent agglomeration in the boat test. Run BR-P75-2, made at the same amount of O_2 consumption, but over a 60-minute period, was free flowing when a sample was subjected to the boat test. The volatile matter and sample weight loss were greater in this test than in the first. In the third test, BR-P75-3, the residence time was maintained at 60 minutes, but the O_2 consumption reduced to 1 SCF/lb of coal. Although the volatile matter and weight loss were similar to that of BR-P75-2, the treatment was not sufficient, as there was some agglomeration when a sample was boat tested. Based on these data, the necessary pretreatment conditions for this coal are 1 hour residence at 750°F with 2 SCF O_2 consumed per pound of coal.

The results for the Pennsylvania-mine, Pittsburgh-seam coal pretreatment are presented in Table 8. Procedures for heating and air introduction were the same as with the other coal. Run BR-P75-4 was made with 1 SCF O_2 over a 30-minute period. Runs BR-P75-5 and BR-P75-6 were made with 2 SCF O_2/lb of coal with residence times of 30 minutes and 60 minutes, respectively. All of these samples were non-agglomerating in the boat test. This coal has a very high ash and a lower volatile content than the West Virginia coal from the same nominal seam. It is, therefore, easier to pretreat, and conditions of 1 SCF O_2/lb of coal over 30 minutes residence time should be sufficient to render it noncaking.

TABLE 7. ANALYSIS FOR PRETREATMENT OF PITTSBURGH SEAM (W. VA.) COAL

	Coal	BR-P75-1	BR-P75-2	BR-P75-3
Proximate Analysis wt%				
Moisture	2.2	0.5	0.3	0.5
Volatile Matter	35.9	27.7	25.5	25.7
Ash	10.6	11.4	13.0	12.6
Fixed Carbon	51.3	60.4	61.2	61.2
Total	100.0	100.0	100.0	100.0
Ultimate Analysis wt% (Dry)				
Ash	10.87	11.43	13.07	12.62
Carbon	73.40	72.70	71.50	72.30
Hydrogen	4.87	4.14	3.93	4.08
Sulfur	2.77	2.26	2.36	2.44
Nitrogen	1.37	1.46	1.42	1.42
Oxygen	6.72	8.01	7.72	7.14
Total	100.00	100.00	100.00	100.00
Weight Loss % (Dry Basis)		12.9	16.5	16.2
Gross Heating Value Btu/lb	13,185	12,589	12,294	12,491
Boat Agglomeration Test		Agglomerated	Free Flowing	Slight Agglomeration
O_2/lb Coal, SCF		2	2	1
Time at 750°F Min		30	60	60

TABLE 8. ANALYSIS FOR PRETREATMENT OF PITTSBURGH SEAM (PENN.) COAL

	Coal	BR-P75-4	BR-P75-5	BR-P75-6
Proximate Analysis wt%				
Moisture	3.0	0.3	0.6	0.5
Volatile Matter	26.0	19.7	18.3	18.8
Ash	33.3	35.3	36.2	37.7
Fixed Carbon	37.7	44.7	44.9	43.0
Total	100.0	100.0	100.0	100.0
Ultimate Analysis wt% (Dry)				
Ash	34.34	35.41	36.44	37.94
Carbon	52.50	52.10	50.90	49.20
Hydrogen	3.54	2.94	2.69	2.78
Sulfur	1.35	1.23	1.11	1.13
Nitrogen	1.08	1.13	1.14	1.08
Oxygen	7.19	7.10	7.72	7.87
Total	100.00	100.00	100.00	100.00
Weight Loss % (Dry Basis)		13.7	16.5	16.7
Gross Heating Value Btu/lb	9,208	8,830	8,506	8,262
Boat Agglomeration Test		Free Flowing	Free Flowing	Free Flowing
O_2/lb Coal SCF		1	2	2
Time at 750°F, Min		30	30	60

COAL PREPARATION – CONCLUSIONS

Coal-crushing and pretreatment testing have proved:

- *A coal-crushing circuit can be designed to minimize fines production and hence elutriation losses.*
- *Eastern coals can be satisfactorily pretreated so that they are non-agglomerating in the fluidized-bed hydrotreatment process unit.*

LABORATORY-SCALE TEST WORK – HYDRODESULFURIZATION

WESTERN KENTUCKY NO. 9 COAL

A series of thermobalance tests was made with the pretreated Western Kentucky No. 9 coal. The thermobalance feed material was screened at 40 mesh so material would not be lost through the stainless mesh basket. Analyses for the coal, pretreated coal, and +40 mesh pretreated coal are listed in Table 9. Apparently the sulfur is not evenly distributed in the different size fractions of the coal, and "equivalent" sulfur values in the raw and pretreated coal were back-calculated for the +40 mesh fraction for data analysis.

The 10 thermobalance run results are shown in Table 10. Coal and pretreated coal values have been back-calculated to reflect +40 mesh sulfur contents. Calculations for the table were based on 100 lb of coal (dry basis) before pretreatment, yielding 90.84 lb after pretreatment. The pretreated coal, when mixed, is blended with lime in a 2:1 lime/coal ratio by weight. Weights after testing are the amounts recoverable from 100 lb of dry coal feedstock. The final sulfur percentages in the reduced data assume sulfide and sulfate removal by washing or mechanical means.

Tests were run in pairs, with identical conditions imposed: the first with a coal and lime mix and the second with coal only. The first two tests (TB-76-1 and TB-76-2) were heated at 5°F/min to 1300°F and held at that temperature for 30 minutes. The sulfur values in the treated coal are not significantly different at 0.82% and 0.76% or in sulfur-by-type distribution. Considerably more treated coal is recovered in the no-lime test (65.40%) than in the lime test (54.92%).

Tests TB-76-3 and TB-76-4 were heated at 5°F/min to 1400°F and were held for 30 minutes. Total sulfur and by-type distribution are similar in these tests, with the no-lime test showing slightly better reduction. As in the first pair of tests, treated coal recovery is more in the no-lime test.

The 5°F/min rate was used in TB-76-5 and TB-76-6, also. Final temperature was 1500°F with no holding time. More sulfur was removed in the test without lime, 97.5%, than in the one with lime, 92.1%. In the lime test, the treated coal recovery is 52.42%, without lime the recovery was 63.54%.

TB-76-7 and TB-76-8 were heated to 1500°F at 5°F/min and held for 30 minutes. Total sulfur percentages are essentially the same. Again, the recovery for the no-lime test is better than the lime test.

A 20°F/min rate to 1500°F was used for TB-76-9 and TB-76-10. These were held for 240 minutes to make the total run time similar to the times of the slower heat-up tests. The longer time at the final temperature results in slightly higher weight losses. Sulfur values are lower in these tests than any of the previous ones, indicating that the temperature and holding time are effective. Recoveries follow the same trend as previous tests.

TABLE 9. WESTERN KENTUCKY NO. 9 COAL ANALYSIS

	Coal	Prètreated Coal	+40 Mesh PR-SP-1
Proximate Analysis, wt %			
Moisture	5.8	0.8	1.2
Volatile Matter	36.3	27.7	26.6
Ash	10.6	11.2	13.9
Fixed Carbon	47.3	60.3	58.3
Total	100.0	100.0	100.0
Ultimate Analysis, wt %			
Ash	11.24	11.25	14.03
Carbon	70.00	71.40	68.60
Hydrogen	4.54	4.06	3.84
Sulfur	3.74	3.16	2.87
Nitrogen	1.53	1.64	1.57
Oxygen	8.95	8.49	9.09
Total	100.00	100.00	100.00

TABLE 10. THERMOBALANCE TEST RUN DATA, PRETREATED WESTERN KENTUCKY NO. 9 COAL

Run No.	Feed Coal*	Pretreated Coal	TB-76-1			TB-76-2	
Feed Ratio, lime/coal			2/1			0/1	
Coal	W. Ky. No. 9	W. Ky. No. 9	Pret. W. Ky. No. 9			Pret. W. Ky. No. 9	
Heating Rate, °F/min			5			5	
Terminal Temperature, °F		750	1300			1300	
Holding Time, min		30	30			30	
			Feed	Float	Sink	Feed	Residue
Sulfur, wt %							
Sulfide	0.02	0.01	0.00	0.28	0.92	0.01	0.26
Sulfate	0.52	0.10	0.03	0.04	0.04	0.10	0.04
Pyritic	0.92	1.25	0.42	0.03	0.00	1.25	0.04
Organic	1.57	1.29	0.43	0.47	0.06	1.29	0.42
Total	3.03	2.65	0.88	0.82	1.02	2.65	0.76
Weight, gms							
Initial	100.00		3.6829			2.5291	
Treated		90.84		0.7502	2.4528		1.8209
Weight Loss, %							
Total, wt		9.16		13.04			28.00
Coal, wt		9.16		39.12			28.00
Reduced Data							
Weight, lb		90.84	272.52	54.92	179.56	90.84	65.40
Sulfur Weight, lb							
Sulfide	0.02	0.01	0.01	0.15	1.65	0.01	0.17
Sulfate	0.52	0.09	0.09	0.02	0.07	0.09	0.03
Pyritic	0.92	1.13	1.13	0.02	0.00	1.13	0.03
Organic	1.57	1.17	1.17	0.26	0.11	1.17	0.27
Total	3.03	2.40	2.40	0.45	1.83	2.40	0.50
Sulfur Content, %							
Sulfide	0.02	0.01	0.00				
Sulfate	0.52	0.10	0.03				
Pyritic	0.92	1.25	0.42	0.03			0.04
Organic	1.57	1.29	0.43	0.47			0.42
Total	3.03	2.65	0.88	0.50			0.46
Sulfur Removal, wt %							
From Feed				88.3			87.5
From Coal		20.5		90.7			90.1

*Calculated for +40 mesh fraction.

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TABLE 10. THERMOBALANCE TEST RUN DATA, PRETREATED WESTERN KENTUCKY NO. 9 COAL (Continued)

Run No.	Feed Coal*	Pretreated Coal*	TB-76-3			TB-76-4	
Feed Ratio, lime/coal			2/1			0/1	
Coal	W. Ky. No. 9	W. Ky. No. 9	Pret. W. Ky. No. 9			Pret. W. Ky. No. 9	
Heating Rate, °F/min			5			5	
Terminal Temperature, °F		750	1400			1400	
Holding Time, min		30	30			30	
			Feed	Float	Sink	Feed	Residue
Sulfur, wt %							
Sulfide	0.02	0.01	0.00	0.29	0.92	0.00	0.23
Sulfate	0.52	0.10	0.03	0.00	0.01	0.10	0.00
Pyritic	0.92	1.25	0.42	0.02	0.00	1.25	0.01
Organic	1.57	1.29	0.43	0.33	0.19	1.29	0.33
Total	3.03	2.65	0.88	0.64	1.12	2.65	0.57
Weight, gms							
Initial	100.00		3.7700			2.4656	
Treated		90.84		0.8550	2.3944		1.7255
Weight Loss, %							
Total, wt		9.16		13.81			30.02
Coal, wt		9.16		41.43			30.02
Reduced Data							
Weight, lb		90.84	272.52	61.80	173.08	90.84	63.57
Sulfur Weight, lb							
Sulfide	0.02	0.01	0.01	0.18	1.59	0.01	0.15
Sulfate	0.52	0.09	0.09	0.00	0.02	0.09	0.00
Pyritic	0.92	1.13	1.13	0.01	0.00	1.13	0.01
Organic	1.57	1.17	1.17	0.20	0.33	1.17	0.21
Total	3.03	2.40	2.40	0.39	1.94	2.40	0.37
Sulfur Content, %							
Sulfide	0.02	0.01	0.00				
Sulfate	0.52	0.10	0.03				
Pyritic	0.92	1.25	0.42	0.02			0.01
Organic	1.57	1.29	0.43	0.33			0.33
Total	3.03	2.65	0.88	0.35			0.34
Sulfur Removal, wt %							
From Feed				91.2			90.8
From Coal		20.5		93.1			92.7

*Calculated for +40 mesh fraction.

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TABLE 10. THERMOBALANCE TEST RUN DATA, PRETREATED WESTERN KENTUCKY NO. 9 COAL (Continued)

Run No.	Feed Coal*	Pretreated Coal*	TB-76-5			TB-76-6	
Feed Ratio, lime/coal			2/1			0/1	
Coal	W. Ky. No. 9	W. Ky. No. 9	Pret. W. Ky. No. 9			Pret. W. Ky. No. 9	
Heating Rate, °F/min			5			5	
Terminal Temperature, °F		750	1500			1500	
Holding Time, min		30	0			0	
			Feed	Float	Sink	Feed	Residue
Sulfur, wt %							
Sulfide	0.02	0.01	0.00	0.10	0.95	0.01	0.24
Sulfate	0.52	0.10	0.03	0.01	0.05	0.10	0.00
Pyritic	0.92	1.25	0.42	0.02	0.00	1.25	0.04
Organic	1.57	1.29	0.43	0.35	0.08	1.29	0.05
Total	3.03	2.65	0.88	0.48	1.08	2.65	0.33
Weight, gms							
Initial	100.00		3.5448			2.2834	
Treated		90.84		0.6819	2.3701		1.5972
Weight Loss, %							
Total, wt		9.16		13.90			30.05
Coal, wt		9.16		41.71			30.05
Reduced Data							
Weight, lb		90.84	272.52	52.42	182.22	90.84	63.54
Sulfur Weight, lb							
Sulfide	0.02	0.01	0.01	0.05	1.73	0.01	0.15
Sulfate	0.52	0.09	0.09	0.01	0.09	0.09	0.00
Pyritic	0.92	1.13	1.13	0.01	0.00	1.13	0.03
Organic	1.57	1.17	1.17	0.18	0.15	1.17	0.03
Total	3.03	2.40	2.40	0.25	1.97	2.40	0.21
Sulfur Content, %							
Sulfide	0.02	0.01	0.01			0.01	
Sulfate	0.52	0.10	0.10			0.10	
Pyritic	0.92	1.25	1.25	0.02		1.25	0.04
Organic	1.57	1.29	1.29	0.35		1.29	0.05
Total	3.03	2.65	2.65	0.37		2.65	0.09
Sulfur Removal, wt %							
From Feed				92.1			97.5
From Coal		20.5		93.7			98.0

*Calculated for +40 mesh fraction.

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TABLE 10. THERMOBALANCE TEST RUN DATA, PRETREATED WESTERN KENTUCKY NO. 9 COAL (Continued)

Run No.	Feed Coal*	Pretreated Coal*	TB-76-7			TB-76-8	
Feed Ratio, lime/coal			2/1			0/1	
Coal	W. Ky. No. 9	W. Ky. No. 9	Pret. W. Ky. No. 9			Pret. W. Ky. No. 9	
Heating Rate, °F/min			5			5	
Terminal Temperature, °F		750	1500			1500	
Holding Time, min		30	30			30	
			Feed	Float	Sink	Feed	Residue
Sulfur, wt %							
Sulfide	0.02	0.01	0.00	0.11	0.84	0.01	0.19
Sulfate	0.52	0.10	0.03	0.00	0.08	0.10	0.00
Pyritic	0.92	1.25	0.42	0.00	0.00	1.25	0.03
Organic	1.57	1.29	0.43	0.40	0.21	1.29	0.28
Total	3.03	2.65	0.88	0.51	1.13	2.65	0.50
Weight, gms							
Initial	100.00		3.4669			2.2871	
Treated		90.84		0.6562	2.3007		1.5766
Weight Loss, %							
Total, wt		9.16		14.71			31.07
Coal, wt		9.16		44.13			31.07
Reduced Data							
Weight, lb		90.84	272.52	51.58	180.84	90.84	62.62
Sulfur Weight, lb							
Sulfide	0.02	0.01	0.01	0.06	1.52	0.01	0.12
Sulfate	0.52	0.09	0.09	0.00	0.14	0.09	0.00
Pyritic	0.92	1.13	1.13	0.00	0.00	1.13	0.02
Organic	1.57	1.17	1.17	0.21	0.38	1.17	0.18
Total	3.03	2.40	2.40	0.27	2.04	2.40	0.32
Sulfur Content, %							
Sulfide	0.02	0.01	0.01				
Sulfate	0.52	0.10	0.10				
Pyritic	0.92	1.25	1.25	0.00			0.03
Organic	1.57	1.29	1.29	0.40			0.28
Total	3.03	2.65	2.65	0.40			0.31
Sulfur Removal, wt %							
From Feed				83.3			87.1
From Coal		20.5		86.8			89.8

*Calculated for +40 mesh fraction.

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TABLE 10. THERMOBALANCE TEST RUN DATA, PRETREATED WESTERN KENTUCKY NO. 9 COAL (Continued)

Run No.	Feed Coal*	Pretreated Coal*	TB-76-9			TB-76-10	
Feed Ratio, lime/coal			2/1			0/1	
Coal	W. Ky. No. 9	W. Ky. No. 9	Pret. W. Ky. No. 9			Pret. W. Ky. No. 9	
Heating Rate, °F/min			20			20	
Terminal Temperature, °F		750	1500			1500	
Holding Time, min		30	240			240	
			Feed	Float	Sink	Feed	Residue
Sulfur, wt %							
Sulfide	0.02	0.01	0.00	0.09	1.27	0.00	0.16
Sulfate	0.52	0.10	0.03	0.01	0.01	0.10	0.00
Pyritic	0.92	1.25	0.42	0.01	0.00	1.25	0.02
Organic	1.57	1.29	0.43	0.25	0.06	1.29	0.15
Total	3.03	2.65	0.88	0.36	1.34	2.65	0.33
Weight, gms							
Initial	100.00		3.6562			2.0635	
Treated		90.84		0.6918	2.3940		1.3886
Weight Loss, %							
Total, wt		9.16		15.60			32.71
Coal, wt		9.16		46.79			32.71
Reduced Data							
Weight, lb		90.84	272.52	51.57	178.44	90.84	61.13
Sulfur Weight, lb							
Sulfide	0.02	0.01	0.01	0.05	2.27	0.01	0.10
Sulfate	0.52	0.09	0.09	0.01	0.02	0.09	0.00
Pyritic	0.92	1.13	1.13	0.01	0.00	1.13	0.01
Organic	1.57	1.17	1.17	0.13	0.11	1.17	0.09
Total	3.03	2.40	2.40	0.20	2.40	2.40	0.20
Sulfur Content, %							
Sulfide	0.02	0.01	0.00			0.00	
Sulfate	0.52	0.10	0.03			0.10	
Pyritic	0.92	1.25	0.42	0.01		1.25	0.02
Organic	1.57	1.29	0.43	0.25		1.29	0.15
Total	3.03	2.65	0.88	0.26		2.65	0.17
Sulfur Removal, wt %							
From Feed				94.2			95.8
From Coal		20.5		95.4			96.7

*Calculated for +40 mesh fraction.

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Sulfur values from the treated material reduced data (Runs TB-76-1 through TB-76-10) are presented graphically in Figure 4. The resultant sulfur contents (pyritic plus organic) are shown as a function of temperature. Sulfur content decreases as the temperature increases and the no-lime tests show consistently better removal than the lime tests.

Figure 5 shows treated coal recovery as a function of the final temperature. Characteristics of the coal, coal volatiles, lime, or the float-sink technique may have resulted in the higher recovery at 1400°F for the mix tests. The no-lime tests show nearly a straight-line decrease in recovery from 1300°F to 1500°F. Recoveries are significantly greater for the no-lime tests than when lime is used; the improved recovery is a factor for future decisions. The probable reason for better recovery is that heavier, higher-ash particles report to the sink (lime fraction of the float-sink separation); also, the volatiles are absorbed by the lime during the test. The end result is a reduced quantity of treated product.

Calculations for SO_2 emissions for each of the 10 tests are shown in Table 11. From previous batch reactor work, the heating value of the float-sink treated coal has been in the range of 10,700 to 11,600 Btu/lb*. The lower figure was used for

TABLE 11. CALCULATED SO_2 EMISSIONS FROM COMBUSTION OF TREATED COAL

	Emission, lb $SO_2/10^6$ Btu	
Test No.	Total Sulfur in Treated Coal	Pyritic and Organic Sulfur Only
TB-76-1	1.53	0.94
TB-76-2	1.42	0.86
TB-76-3	1.20	0.66
TB-76-4	1.07	0.65
TB-76-5	0.90	0.64
TB-76-6	0.62	0.17
TB-76-7	0.95	0.75
TB-76-8	0.93	0.58
TB-76-9	0.67	0.49
TB-76-10	0.62	0.32

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* The small samples available from the thermobalance runs preclude the experimental measurement of heating value in addition to the required sulfur analyses.

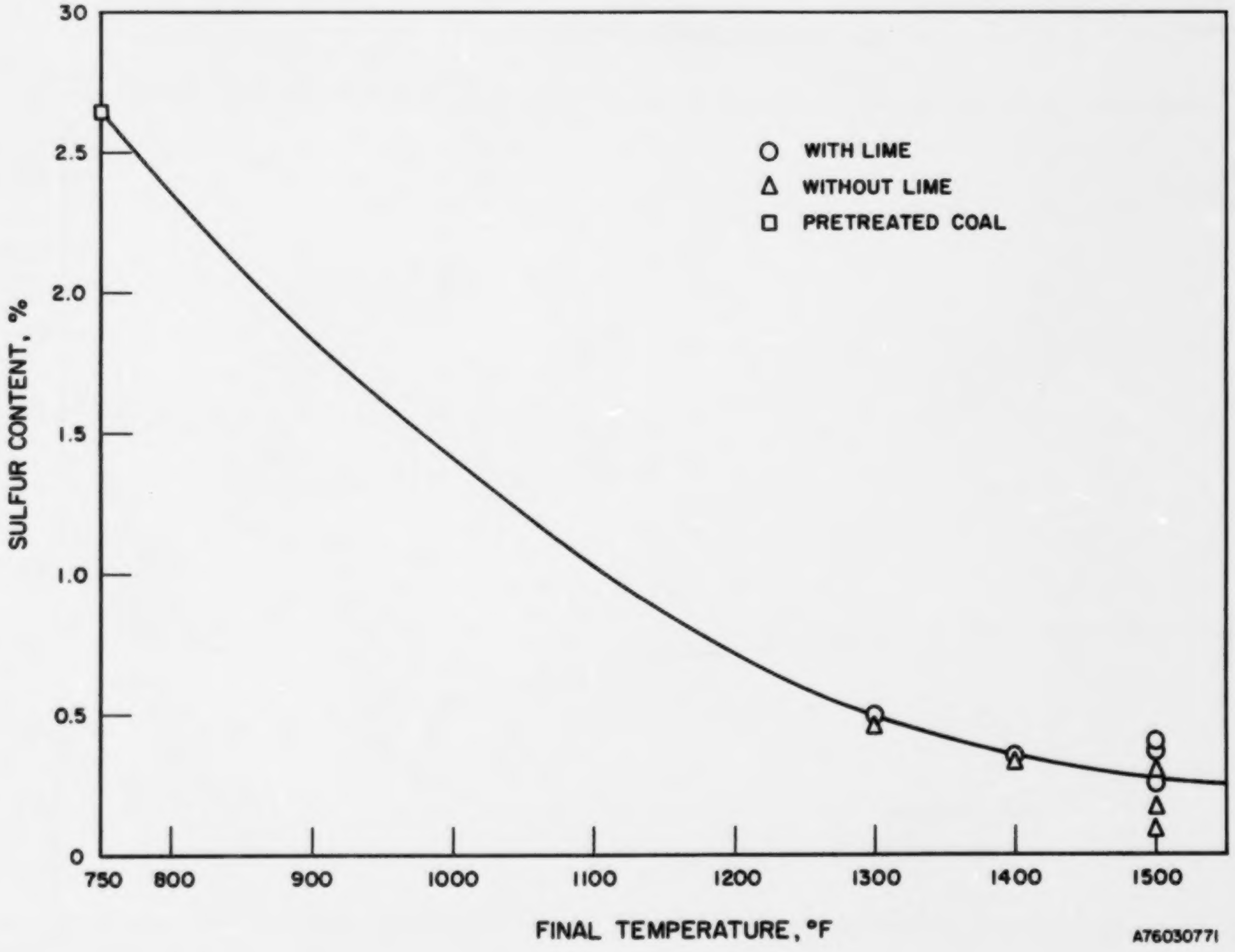

Figure 4. Treated material sulfur content, Western Kentucky No. 9 coal.

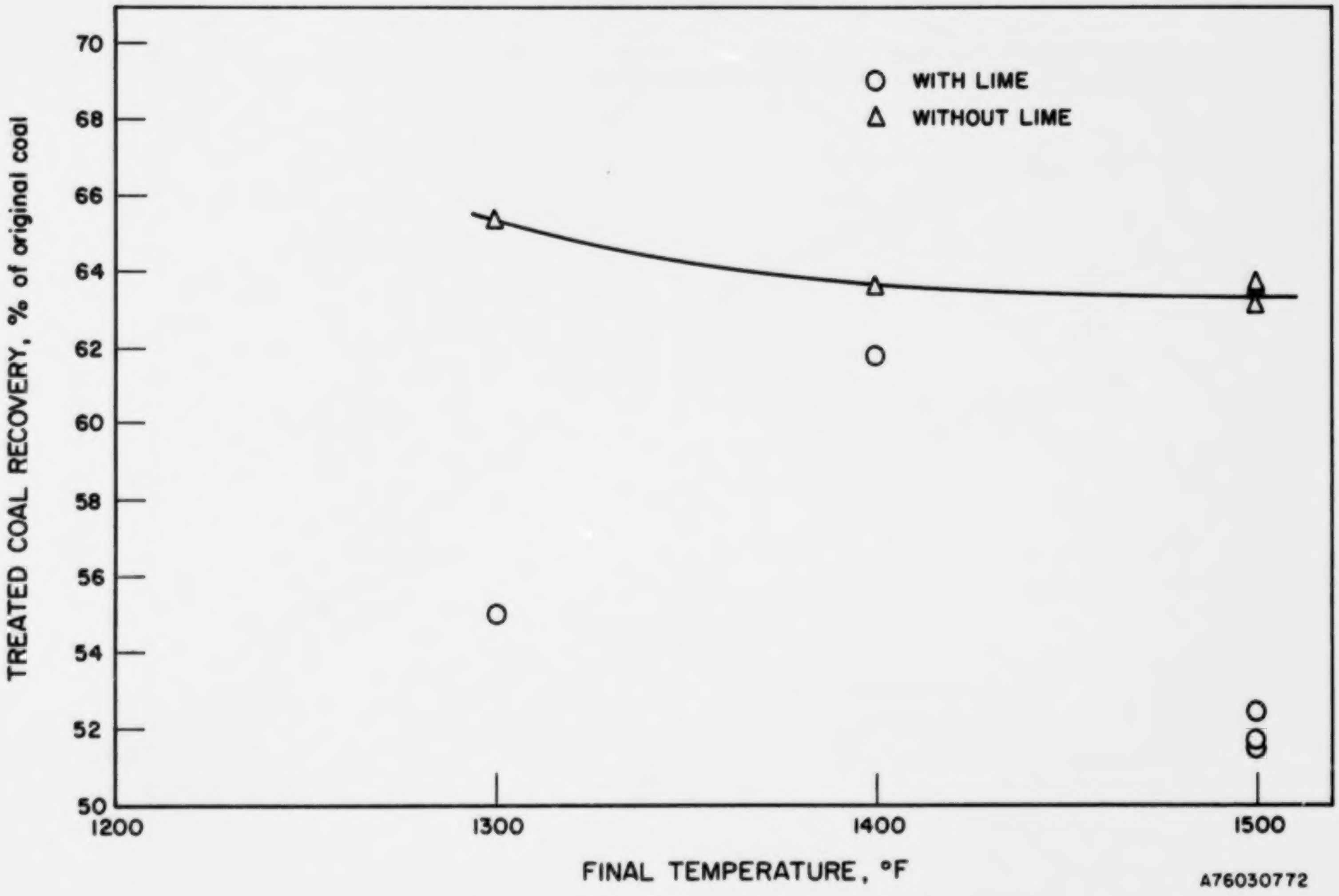

Figure 5. Treated material recovery, Western Kentucky No. 9 coal.

these calculations, resulting in conservatively estimated emissions. All the tests are below the 1.2 lb SO_2/million Btu if the sulfide and sulfate can be removed as indicated. Tests at temperatures of 1400°F and above show acceptable SO_2 emissions even without removal of the sulfide and sulfate. More definitive values will be obtained from the batch reactor runs as enough sample is available to determine exact heating values and the SO_2 emissions from them.

PITTSBURGH SEAM – WEST VIRGINIA COAL

An identical series of tests was conducted with the Pittsburgh-seam coal from the West Virginia mine. Table 12 lists the analyses for the original coal, pretreated coal, and +40 mesh pretreated coal.

Results from the 10 thermobalance tests are listed in Table 13. Sulfur is not distributed equally in the different size fractions, so equivalent values are calculated for the original and pretreated coals. Values are based on 100 lb of dry coal before pretreatment, which yields 83.5 lb of pretreated material. In tests using lime, the mixture is 2:1 lime/coal by weight. Final sulfur percentages for the reduced data assume sulfide and sulfate removal by chemical or mechanical means.

The tests were run in pairs, at identical conditions, one with pretreated coal only and one with the mixture. In this way, the value of the lime addition can be measured. Heat-up rates, terminal temperatures, and holding times at the temperatures are shown in Table 13.

Comparison of the pairs of tests shows slightly better total sulfur removal in the lime tests than in the no-lime tests. However, in the reduced data, assuming sulfide and sulfate removal, the sulfur values are nearly the same for the float or residue of each pair. The sample from TB-76-14 was accidentally lost during the organic sulfur determination, and the test was not rerun because of the consistency of the other test results.

The sums of the pyritic and organic sulfur values from the reduced data are shown in Figure 6. Sulfur content is reduced as the temperature increases or with longer holding time at the final temperature.

Treated material recovery is shown in Figure 7. The no-lime tests exhibit better recovery at all temperatures. A higher recovery for the lime test at 1400°F over the other lime tests is the same as experienced with the pretreated Western Kentucky No. 9 coal. Perhaps this effect is an interaction of coal, lime, and coal volatiles at this temperature.

Calculations for SO_2 emission from the treated coal samples are listed in Table 14. The pretreated coal has a heating of 12,300 Btu/lb, and a similar value for the final product was assumed. If only the pyritic and organic type sulfurs are considered, all tests have acceptable values. Considering the total sulfur, only the tests at 1300°F are too high in SO_2 emission; the rest are below the 1.2 lb/million Btu limit.

TABLE 12. PITTSBURGH SEAM, WEST VIRGINIA COAL ANALYSIS

	Coal	Pretreated Coal	Pretreated +40 Mesh Coal
Proximate Analysis, wt %			
Moisture	2.2	0.3	0.7
Volatile Matter	35.9	25.5	26.6
Ash	10.6	13.0	9.8
Fixed Carbon	51.3	61.2	62.9
Total	100.0	100.0	100.0
Ultimate Analysis, wt %			
Ash	10.87	13.07	9.83
Carbon	73.40	71.5	74.80
Hydrogen	4.87	3.93	4.20
Sulfur	2.77	2.36	2.16
Nitrogen	1.37	1.42	1.47
Oxygen	6.72	7.72	7.54
Total	100.00	100.00	100.00

TABLE 13. THERMOBALANCE TEST RUN DATA, PRETREATED PITTSBURGH SEAM, WEST VIRGINIA COAL

Run No.	Coal*	Pretreatment*	TB-76-11			TB-76-12	
Feed Ratio, lime/coal			2:1			0:1	
Coal	Pitt. Seam, W. Va.		Pret. Pitt. Seam, W. Va.				
Heating Rate, °F/min			5			5	
Terminal Temperature, °F		750	1300			1300	
Holding Time, min		60	30			30	
Sulfur, wt % as			Feed	Float	Sink	Feed	Residue
Sulfide	0.00	0.00	0.00	0.06	0.16	0.00	0.26
Sulfate	0.42	0.27	0.09	0.05	0.35	0.27	0.00
Pyritic	0.63	0.37	0.12	0.01	0.00	0.37	0.02
Organic	1.36	1.47	0.49	0.63	0.08	1.47	0.60
Total	2.41	2.11	0.70	0.75	0.59	2.11	0.88
Weight, g							
Initial	100		4.3576			2.1626	
Treated		83.5		1.0014	2.7296		1.5985
Weight Loss, %							
Total Weight		16.5		14.26			26.08
Coal Weight		16.5		42.80			26.08
Reduced Data							
Weight, lb		83.5	250.50	57.65	157.13	83.50	61.72
Sulfur Weight, lb							
Sulfide	0.00	0.00	0.00	0.03	0.25	0.00	0.16
Sulfate	0.42	0.23	0.23	0.03	0.55	0.23	0.00
Pyritic	0.63	0.31	0.31	0.01	0.00	0.31	0.01
Organic	1.36	1.23	1.23	0.36	0.13	1.23	0.37
Total	2.41	1.77	1.77	0.44	0.93	1.77	0.54
Sulfur Content, %							
Sulfide	0.00	0.00	0.00			0.00	
Sulfate	0.42	0.27	0.09			0.27	
Pyritic	0.63	0.37	0.12	0.01		0.37	0.02
Organic	1.36	1.47	0.49	0.63		1.47	0.60
Total	2.41	2.11	0.70	0.64		2.11	0.62
Sulfur Removal, wt %							
From Feed				79.9			78.5
From Coal				84.6			84.2

*Calculated for 40 mesh fraction.

TABLE 13. THERMOBALANCE TEST RUN DATA, PRETREATED PITTSBURGH SEAM, WEST VIRGINIA COAL (Continued)

Run No.	Coal*	Pretreatment*	TB-76-13			TB-76-14	
Feed Ratio, lime/coal				2:1		0:1	
Coal	Pitt. Seam, W. Va.		Pret. Pitt. Seam, W. Va.				
Heating Rate, °F/min				5		5	
Terminal Temperature, °F		750		1400		1400	
Holding Time, min		60		30		30	
Sulfur, wt % as			Feed	Float	Sink	Feed	Residue
Sulfide	0.00	0.00	0.00	0.12	0.45	0.00	0.26
Sulfate	0.42	0.27	0.09	0.02	0.05	0.27	0.01
Pyritic	0.63	0.37	0.12	0.00	0.00	0.37	0.01
Organic	1.36	1.47	0.49	0.41	0.02	1.47	——
Total	2.41	2.11	0.70	0.55	0.52	2.11	
Weight, g							
Initial	100		4.4926			2.3176	
Treated		83.5		1.0527	2.8916		1.6680
Weight Loss, %							
Total Weight		16.5		12.20			28.03
Coal Weight		16.5		36.61			28.03
Reduced Data							
Weight, lb		83.5	250.50	58.70	161.24	83.50	60.09
Sulfur Weight, lb							
Sulfide	0.00	0.00	0.00	0.07	0.73	0.00	0.16
Sulfate	0.42	0.23	0.23	0.01	0.08	0.23	0.01
Pyritic	0.63	0.31	0.31	0.00	0.00	0.31	0.01
Organic	1.36	1.23	1.23	0.24	0.03	1.23	——
Total	2.41	1.77	1.77	0.32	0.84	1.77	
Sulfur Content, %							
Sulfide	0.00	0.00	0.00			0.00	
Sulfate	0.42	0.27	0.09			0.27	
Pyritic	0.63	0.37	0.12	0.00		0.37	0.01
Organic	1.36	1.47	0.49	0.41		1.47	--
Total	2.41	2.11	0.70	0.41		2.11	--
Sulfur Removal, wt %							
From Feed				86.4			
From Coal				90.0			

*Calculated for 40 mesh fraction.

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TABLE 13. THERMOBALANCE TEST RUN DATA, PRETREATED PITTSBURGH SEAM, WEST VIRGINIA COAL (Continued)

Run No.	Coal	Pretreatment	TB-76-15			TB-76-16	
Feed Ratio, lime/coal				2:1		0:1	
Coal	Pitt. Seam, W. Va.	Pitt. Seam, W. Va.	Pret. Pitt. Seam, W. Va.	Pret. Pitt. Seam, W. Va.	Pret. Pitt. Seam, W. Va.	Pret. Pitt. Seam, W. Va.	Pret. Pitt. Seam, W. Va.
Heating Rate, ^{o}F/min				5		5	
Terminal Temperature, ^{o}F		750		1500		1500	
Holding Time, min		60		0		0	
			Feed	Float	Sink	Feed	Residue
Sulfur, wt % as							
Sulfide	0.00	0.00	0.00	0.13	0.54	0.00	0.18
Sulfate	0.42	0.27	0.09	0.01	0.02	0.27	0.02
Pyritic	0.63	0.37	0.12	0.01	0.00	0.37	0.01
Organic	1.36	1.47	0.49	0.45	0.03	1.47	0.45
Total	2.41	2.11	0.70	0.60	0.59	2.11	0.66
Weight, g							
Initial	100		4.5513			2.4397	
Treated		83.5		1.0451	2.9484		1.7610
Weight Loss, %							
Total Weight		16.5		12.26			27.82
Coal Weight		16.5		36.77			27.82
Reduced Data							
Weight, lb		83.5	250.50	57.52	162.27	83.50	60.27
Sulfur Weight, lb							
Sulfide	0.00	0.00	0.00	0.07	0.88	0.00	0.11
Sulfate	0.42	0.23	0.23	0.01	0.03	0.23	0.01
Pyritic	0.63	0.31	0.31	0.01	0.00	0.31	0.01
Organic	1.36	1.23	1.23	0.26	0.05	1.23	0.27
Total	2.41	1.77	1.77	0.35	0.96	1.77	0.40
Sulfur Content, %							
Sulfide	0.00	0.00	0.00			0.00	
Sulfate	0.42	0.27	0.42			0.27	
Pyritic	0.63	0.37	0.63	0.01		0.37	0.01
Organic	1.36	1.47	1.36	0.45		1.47	0.45
Total	2.41	2.11	2.41	0.46		2.11	0.46
Sulfur Removal, wt %							
From Feed				85.3			84.2
From Coal				88.8			88.4

*Calculated for 40 mesh fraction.

TABLE 13. THERMOBALANCE TEST RUN DATA, PRETREATED PITTSBURGH SEAM, WEST VIRGINIA COAL (Continued)

Run No.	Coal*	Pretreatment*	TB-76-17			TB-76-18	
Feed Ratio, lime/coal			2:1			0:1	
Coal	Pitt. Seam, W. Va.		Pret. Pitt. Seam, W. Va.				
Heating Rate, °F/min			5			5	
Terminal Temperature, °F		750	1500			1500	
Holding Time, min		60	30			30	
Sulfur, wt % as			Feed	Float	Sink	Feed	Residue
Sulfide	0.00	0.00	0.00	0.13	0.56	0.00	0.29
Sulfate	0.42	0.27	0.09	0.02	0.02	0.27	0.00
Pyritic	0.63	0.37	0.12	0.02	0.00	0.37	0.01
Organic	1.36	1.47	0.49	0.33	0.04	1.47	0.39
Total	2.41	2.11	0.70	0.50	0.62	2.11	0.69
Weight, g							
Initial	100		4.5277			2.4268	
Treated		83.5		1.0010	2.9443		1.7168
Weight Loss, %							
Total Weight		16.5		12.86			29.26
Coal Weight		16.5		38.59			29.26
Reduced Data							
Weight, lb		83.5	250.50	55.38	162.91	83.50	59.10
Sulfur Weight, lb							
Sulfide	0.00	0.00	0.00	0.08	0.91	0.00	0.17
Sulfate	0.42	0.23	0.23	0.01	0.03	0.23	0.00
Pyritic	0.63	0.31	0.31	0.01	0.00	0.31	0.01
Organic	1.36	1.23	1.23	0.18	0.07	1.23	0.23
Total	2.41	1.77	1.77	0.28	1.01	1.77	0.41
Sulfur Content, %							
Sulfide	0.00	0.00	0.00			0.00	
Sulfate	0.42	0.27	0.42			0.27	
Pyritic	0.63	0.37	0.63	0.02		0.37	0.01
Organic	1.36	1.47	1.36	0.33		1.47	0.39
Total	2.41	2.11	2.41	0.35		2.11	0.40
Sulfur Removal, wt %							
From Feed				89.3			86.4
From Coal				92.1			90.0

*Calculated for 40 mesh fraction.

TABLE 13. THERMOBALANCE TEST RUN DATA, PRETREATED PITTSBURGH SEAM, WEST VIRGINIA COAL (Continued)

Run No.	Coal*	Pretreatment*	TB-76-19			TB-76-20	
Feed Ratio, lime/coal				2:1		0:1	
Coal	Pitt. Seam, W. Va.		Pret. Pitt. Seam, W. Va.				
Heating Rate, °F/min				20		20	
Terminal Temperature, °F		750		1500		1500	
Holding Time, min		60		240		240	
Sulfur, wt % as			Feed	Float	Sink	Feed	Residue
Sulfide	0.00	0.00	0.00	0.09	0.52	0.00	0.35
Sulfate	0.42	0.27	0.09	0.00	0.03	0.27	0.00
Pyritic	0.63	0.37	0.12	0.02	0.00	0.37	0.02
Organic	1.36	1.47	0.49	0.30	0.10	1.47	0.32
Total	2.41	2.11	0.70	0.41	0.65	2.11	0.69
Weight, g							
Initial	100		4.5337			2.4042	
Treated		83.5		0.9449	2.9796		1.6607
Weight Loss, %							
Total Weight		16.5		13.44			30.93
Coal Weight		16.5		40.31			30.93
Reduced Data							
Weight, lb		83.5	250.50	52.21	164.62	83.50	57.67
Sulfur Weight, lb							
Sulfide	0.00	0.00	0.00	0.05	0.86	0.00	0.20
Sulfate	0.42	0.23	0.23	0.00	0.05	0.23	0.00
Pyritic	0.63	0.31	0.31	0.01	0.00	0.31	0.01
Organic	1.36	1.23	1.23	0.16	0.16	1.23	0.18
Total	2.41	1.77	1.77	0.22	1.07	1.77	0.39
Sulfur Content, %							
Sulfide	0.00	0.00	0.00			0.00	
Sulfate	0.42	0.27	0.42			0.27	
Pyritic	0.63	0.37	0.63	0.02		0.37	0.02
Organic	1.36	1.47	1.36	0.30		1.47	0.32
Total	2.41	2.11	2.41	0.32		2.11	0.34
Sulfur Removal, wt %							
From Feed				90.4			89.3
From Coal				92.9			92.1

*Calculated for 40 mesh fraction.

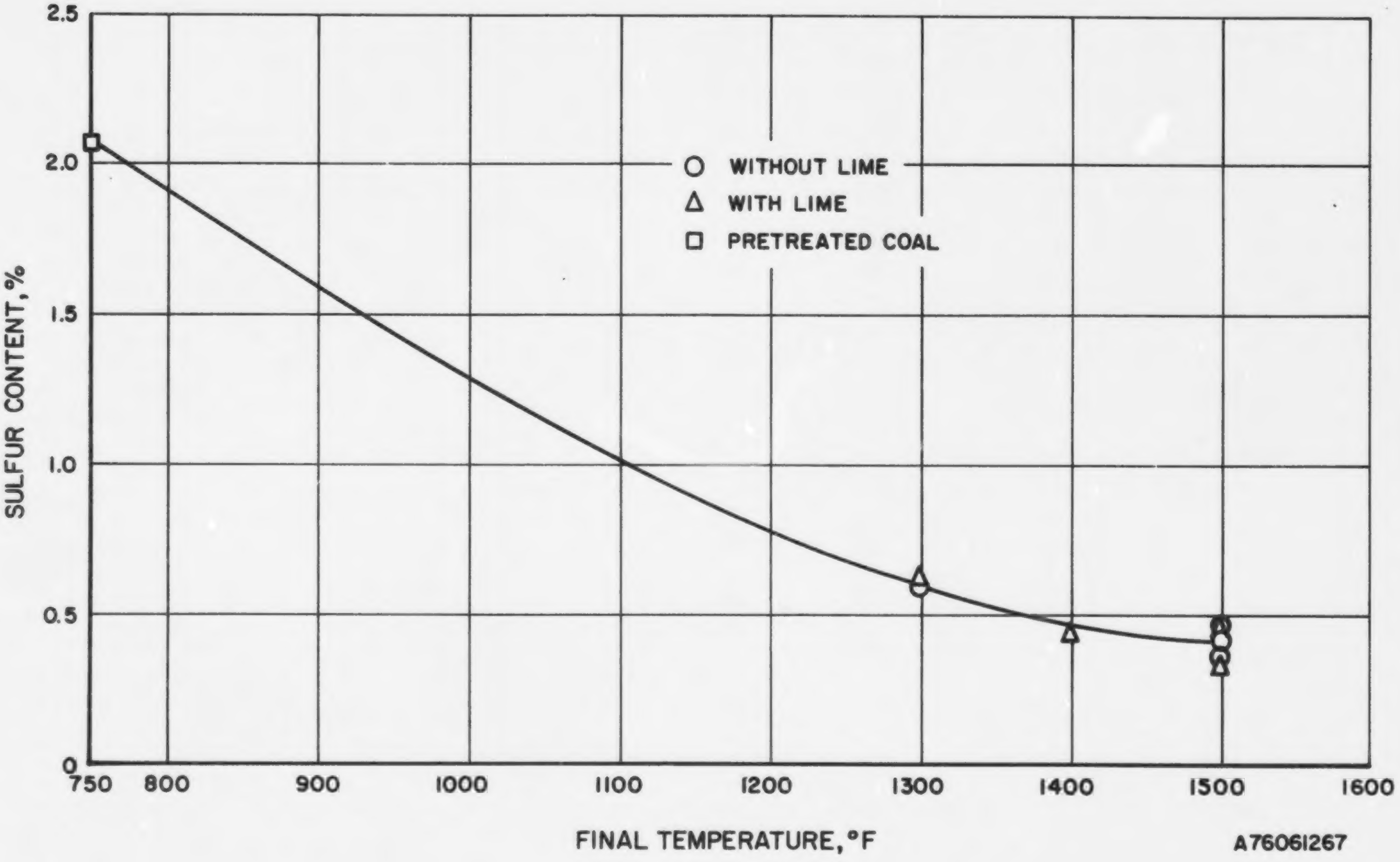

Figure 6. Treated material sulfur content, Pittsburgh seam, West Virginia coal.

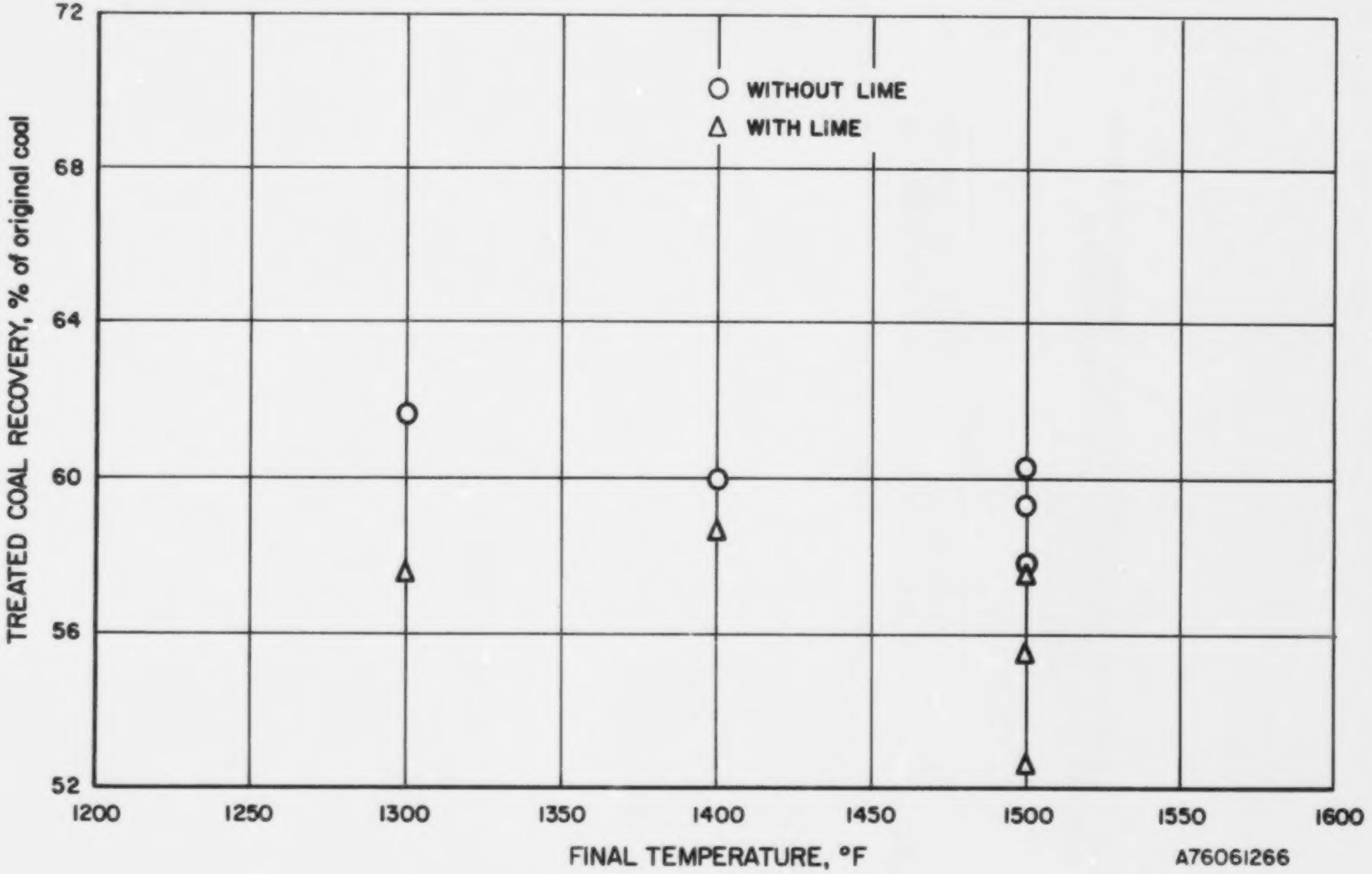

Figure 7. Treated material recovery, Pittsburgh seam, West Virginia coal.

TABLE 14. CALCULATED SULFUR DIOXIDE EMISSIONS FROM COMBUSTION OF TREATED PITTSBURGH SEAM, WEST VIRGINIA COAL

	Emission	
Test No.	Total Sulfur in Treated Coal	Pyritic and Organic Sulfur Only
	lb $SO_2/10^6$ Btu	
TB-76-11	1.22	1.04
TB-76-12	1.43	1.00
TB-76-13	0.89	0.67
TB-76-14	–	–
TB-76-15	0.98	0.75
TB-76-16	1.07	0.75
TB-76-17	0.81	0.57
TB-76-18	1.12	0.65
TB-76-19	0.67	0.52
TB-76-20	1.12	0.55

PITTSBURGH SEAM - PENNSYLVANIA COAL

A series of thermobalance tests was made with the pretreated Pittsburgh-seam coal (Pennsylvania mine). The original coal, pretreated coal, and +40 mesh pretreated coal analyses are shown in Table 15. The coal was again screened at +40 mesh for these tests to prevent loss through the thermobalance basket.

Thermobalance run results are listed in Table 16. Unequal sulfur distribution in the size fractions requires back-calculation of pseudo-sulfur contents for the coal and pretreated coal. The bases for calculations are 100 lb of dry coal before pretreatment and 86.34 lb after pretreatment. Final sulfur values, in the reduced data, assume sulfide and sulfate removal by mechanical or chemical means. The tests were run in pairs: one test with pretreated coal only and one with a lime-pretreated coal mix to assess the value of the lime usage. Other test conditions are as listed in Table 16.

Comparisons of the test pairs for the lime and no-lime operation, Figure 8, show little or no difference in the total weight of sulfur removed. The reduced data treated-material sulfur percentages are lower for the no-lime tests than for the lime tests. Material recovery (Figure 9), however, is higher for the no-lime tests. Sulfur reduction, in general, is greater as the temperature increases. The holding time at 1500°F did not appreciably reduce sulfur content.

The calculated SO_2 emissions from coal that would be treated the same as these samples are presented in Table 17. The small sample does not permit evaluation of the heating value for each test. The assumed heating value of 9150 Btu/lb was estimated from the known values for the coal and pretreated coal. All the treated material samples are below the Federal EPA limit of 1.2 lb SO_2/million Btu, even when considering total sulfur. The original sulfur content of the coal is low and the treatment requirements, therefore, are not as severe as with the other coals.

ILLINOIS NO. 6 COAL

A similar series of tests was made with pretreated Illinois No. 6 coal. Table 18 shows the analyses for the original coal, pretreated coal, and +40 mesh coal.

Results of the 10 tests are listed in Table 19. The quantities are calculated as described previously. Values are based on 100 lb of dry raw coal, before pretreatment, and 90.4 lb after pretreatment.

Analysis of the data shows the lime tests, Figure 10, have a slightly lower sulfur content for the treated material. Sulfur removal for this coal is approximately the same throughout the temperature range. Some benefit is exhibited in a long holding time at 1500°F. Material recovery, Figure 11, for the no-lime tests is better.

The calculated SO_2 emissions for these tests are given in Table 20. A heating value was estimated at 12,500 Btu/lb. All of the calculated values, both total sulfur and pyritic plus organic sulfur, have emission values below the established limits.

TABLE 15. ANALYSES OF PITTSBURGH SEAM COAL (PENNSYLVANIA MINE)

	Coal	Pretreated Coal	+40 Mesh Pretreated Coal
		wt %	
Proximate Analysis			
Moisture	3.0	0.3	0.9
Volatile Matter	26.0	19.7	21.2
Ash	33.3	35.3	33.7
Fixed Carbon	37.7	44.7	44.2
Total	100.0	100.0	100.0
Ultimate Analysis (Dry)			
Ash	34.34	35.41	33.99
Carbon	52.50	52.10	54.10
Hydrogen	3.54	2.94	3.26
Sulfur	1.35	1.23	1.11
Nitrogen	1.08	1.13	1.10
Oxygen	7.19	7.19	6.44
Total	100.00	100.00	100.00

TABLE 16. THERMOBALANCE RUN DATA, PITTSBURGH SEAM COAL (PENNSYLVANIA MINE)

Run No.	Feed Coal*	Pretreated Coal*	TB-76-25			TB-76-26	
Lime/Coal Feed Ratio			2:1			0:1	
Heating Rate, °F/min			5			5	
Terminal Temperature, °F		750	1300			1300	
Holding Time, min		30	30			30	
Sulfur, wt %			Feed	Float	Sink	Feed	Residue
Sulfide	0.01	0.00	0.00	0.03	0.19	0.00	0.14
Sulfate	0.44	0.34	0.11	0.01	0.02	0.34	0.02
Pyritic	0.21	0.16	0.05	0.02	0.00	0.16	0.01
Organic	0.35	0.41	0.14	0.32	0.04	0.41	0.21
Total	1.01	0.91	0.30	0.38	0.25	0.91	0.38
Weight, g							
Initial	100		6.1748			2.6015	
Treated		86.34		0.9513	4.5558		2.0446
Weight Loss, %							
Total Weight		13.66		— 10.81 —			21.41
Coal Weight		13.66		— 32.44 —			21.41
Reduced Data							
Weight, lb			259.02	39.91	191.11	86.34	67.86
Sulfur Weight, lb							
Sulfide	0.01	0.00	0.00	0.01	0.36	0.00	0.10
Sulfate	0.44	0.29	0.28	0.00	0.04	0.28	0.01
Pyritic	0.21	0.14	0.13	0.01	0.00	0.13	0.01
Organic	0.35	0.35	0.36	0.13	0.08	0.36	0.14
Total	1.01	0.78	0.77	0.15	0.48	0.77	0.26
Sulfur Content, %							
Sulfide	0.01	0.00	0.00	0.00		0.00	0.00
Sulfate	0.44	0.34	0.11	0.00		0.34	0.00
Pyritic	0.21	0.16	0.05	0.02		0.16	0.01
Organic	0.35	0.41	0.14	0.32		0.41	0.21
Total	1.01	0.91	0.30	0.34		0.91	0.22
Sulfur Removal, wt %							
From Feed				81.8			80.5
From Coal		22.8		86.1			85.1

* Calculated for 40 mesh fraction

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TABLE 16. THERMOBALANCE RUN DATA, PITTSBURGH SEAM COAL (PENNSYLVANIA MINE) (Continued)

	Feed Coal[a]	Pretreated Coal[a]					
Run No.			TB-76-27			TB-76-28	
Lime/Coal Feed Ratio			2:1			0:1	
Heating Rate, °F/min			5			5	
Terminal Temperature, °F		750	1400			1400	
Holding Time, min		30	30			30	
			Feed	Float	Sink	Feed	Residue
Sulfur, wt %							
Sulfide	0.01	0.00	0.00	0.10	0.23	0.00	0.06
Sulfate	0.44	0.34	0.11	0.00	0.01	0.34	0.04
Pyritic	0.21	0.16	0.05	0.02	0.00	0.16	0.03
Organic	0.35	0.41	0.14	0.24	0.02	0.41	0.22
Total	1.01	0.91	0.30	0.36	0.26	0.91	0.35
Weight, g							
Initial	100		4.4885			2.9990	
Treated		86.34		0.8027	3.2067		2.3440
Weight Loss, %							
Total Weight		13.66	— 10.67 —				21.84
Coal Weight		13.66	— 32.02 —				21.84
Reduced Data							
Weight, lb			259.02	46.32	185.05	86.34	67.48
Sulfur Weight, lb							
Sulfide	0.01	0.00	0.00	0.05	0.42	0.00	0.04
Sulfate	0.44	0.29	0.28	0.00	0.02	0.28	0.03
Pyritic	0.21	0.14	0.13	0.01	0.00	0.13	0.02
Organic	0.35	0.35	0.36	0.11	0.04	0.36	0.15
Total	1.01	0.78	0.77	0.17	0.48	0.77	0.24
Sulfur Content, %							
Sulfide	0.01	0.00	0.00	0.00		0.00	0.00
Sulfate	0.44	0.34	0.11	0.00		0.34	0.00
Pyritic	0.21	0.16	0.05	0.02		0.16	0.03
Organic	0.35	0.41	0.14	0.24		0.41	0.22
Total	1.01	0.91	0.30	0.26		0.91	0.25
Sulfur Removal, wt %							
From Feed				84.4			77.9
From Coal		22.8		88.1			83.2

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[a] Calculated for 40 mesh fraction

TABLE 16. THERMOBALANCE RUN DATA, PITTSBURGH SEAM COAL (PENNSYLVANIA MINE) (Continued)

Run No.	Feed Coal*	Pretreated Coal*	TB-76-29			TB-76-30	
			Feed	Float	Sink	Feed	Residue
Lime/Coal Feed Ratio			2:1			0:1	
Heating Rate, °F/min			5			5	
Terminal Temperature, °F		750	1500			1500	
Holding Time, min		30	0			0	
Sulfur, wt %							
Sulfide	0.01	0.00	0.00	0.05	0.18	0.00	0.22
Sulfate	0.44	0.34	0.11	0.02	0.03	0.34	0.00
Pyritic	0.21	0.16	0.05	0.01	0.00	0.16	0.00
Organic	0.35	0.41	0.14	0.19	0.01	0.41	0.16
Total	1.01	0.91	0.30	0.27	0.22	0.91	0.38
Weight, g							
Initial	100		4.5260			3.2010	
Treated		86.34		0.8005	3.2325		2.4776
Weight Loss, %							
Total Weight		13.66		10.89			22.60
Coal Weight		13.66		32.68			22.60
Reduced Data							
Weight, lb			259.02	45.81	184.99	86.34	66.83
Sulfur Weight, lb							
Sulfide	0.01	0.00	0.00	0.02	0.33	0.00	0.15
Sulfate	0.44	0.29	0.28	0.01	0.06	0.28	0.00
Pyritic	0.21	0.14	0.13	0.00	0.00	0.13	0.00
Organic	0.35	0.35	0.36	0.09	0.02	0.36	0.10
Total	1.01	0.78	0.77	0.12	0.41	0.77	0.25
Sulfur Content, %							
Sulfide	0.01	0.00	0.00	0.00		0.00	0.00
Sulfate	0.44	0.34	0.11	0.00		0.34	0.00
Pyritic	0.21	0.16	0.05	0.01		0.16	0.00
Organic	0.35	0.41	0.14	0.19		0.41	0.16
Total	1.01	0.91	0.30	0.20		0.91	0.16
Sulfur Removal, wt %							
From Feed				88.3			87.0
From Coal		22.8		91.1			90.1

* Calculated for 40 mesh fraction

TABLE 16. THERMOBALANCE RUN DATA, PITTSBURGH SEAM COAL (PENNSYLVANIA MINE) (Continued)

Run No.	Feed Coal*	Pretreated Coal*	TB-76-31			TB-76-32	
Lime/Coal Feed Ratio			2:1			0:1	
Heating Rate, °F/min			5			5	
Terminal Temperature, °F		750	1500			1500	
Holding Time, min		30	30			30	
Sulfur, wt %			Feed	Float	Sink	Feed	Residue
Sulfide	0.01	0.00	0.00	0.08	0.23	0.00	0.21
Sulfate	0.44	0.34	0.11	0.01	0.02	0.34	0.00
Pyritic	0.21	0.16	0.05	0.00	0.00	0.16	0.00
Organic	0.35	0.41	0.14	0.18	0.01	0.41	0.12
Total	1.01	0.91	0.30	0.27	0.26	0.91	0.33
Weight, g							
Initial	100		5.0231			3.3107	
Treated		86.34		0.8353	3.6388		2.5341
Weight Loss, %							
Total Weight		13.66		10.93			23.46
Coal Weight		13.66		32.79			23.46
Reduced Data							
Weight, lb			259.02	43.07	187.64	86.34	66.08
Sulfur Weight, lb							
Sulfide	0.01	0.00	0.00	0.03	0.43	0.00	0.14
Sulfate	0.44	0.29	0.28	0.00	0.04	0.28	0.00
Pyritic	0.21	0.14	0.13	0.00	0.00	0.13	0.00
Organic	0.35	0.35	0.36	0.08	0.02	0.36	0.08
Total	1.01	0.78	0.77	0.11	0.49	0.77	0.22
Sulfur Content, %							
Sulfide	0.01	0.00	0.00	0.00		0.00	0.00
Sulfate	0.44	0.34	0.11	0.00		0.34	0.00
Pyritic	0.21	0.16	0.05	0.00		0.16	0.00
Organic	0.35	0.41	0.14	0.18		0.41	0.12
Total	1.01	0.91	0.30	0.18		0.91	0.12
Sulfur Removal, wt %							
From Feed				89.6			89.6
From Coal		22.8		92.1			92.1

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* Calculated for 40 mesh fraction

TABLE 16. THERMOBALANCE RUN DATA, PITTSBURGH SEAM COAL (PENNSYLVANIA MINE) (Continued)

Run No.	Feed Coal*	Pretreated Coal*	TB-76-33			TB-76-34	
Lime/Coal Feed Ratio			2:1			0:1	
Heating Rate, °F/min			20			20	
Terminal Temperature, °F		750	1500			1500	
Holding Time, min		30	240			240	
Sulfur, wt %			Feed	Float	Sink	Feed	Residue
Sulfide	0.01	0.00	0.00	0.06	0.25	0.00	0.21
Sulfate	0.44	0.34	0.11	0.00	0.02	0.34	0.00
Pyritic	0.21	0.16	0.05	0.02	0.00	0.16	0.00
Organic	0.35	0.41	0.14	0.13	0.01	0.41	0.13
Total	1.01	0.91	0.30	0.21	0.28	0.91	0.34
Weight, g							
Initial	100		4.2900			2.8672	
Treated		86.34		0.6934	3.1094		2.1344
Weight Loss, %							
Total Weight		13.66		— 11.36 —			25.56
Coal Weight		13.66		— 34.07 —			25.56
Reduced Data							
Weight, lb			259.02	41.86	187.74	86.34	64.27
Sulfur Weight, lb							
Sulfide	0.01	0.00	0.00	0.03	0.47	0.00	0.13
Sulfate	0.44	0.29	0.28	0.00	0.03	0.28	0.00
Pyritic	0.21	0.14	0.13	0.01	0.00	0.13	0.00
Organic	0.35	0.35	0.36	0.05	0.02	0.36	0.08
Total	1.01	0.78	0.77	0.09	0.52	0.77	0.21
Sulfur Content, %							
Sulfide	0.01	0.00	0.00	0.00		0.00	0.00
Sulfate	0.44	0.34	0.11	0.00		0.34	0.00
Pyritic	0.21	0.16	0.05	0.02		0.16	0.00
Organic	0.35	0.41	0.14	0.13		0.41	0.13
Total	1.01	0.91	0.30	0.15		0.91	0.13
Sulfur Removal, wt %							
From Feed				92.2			89.6
From Coal		22.8		94.1			92.1

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* Calculated for 40 mesh fraction

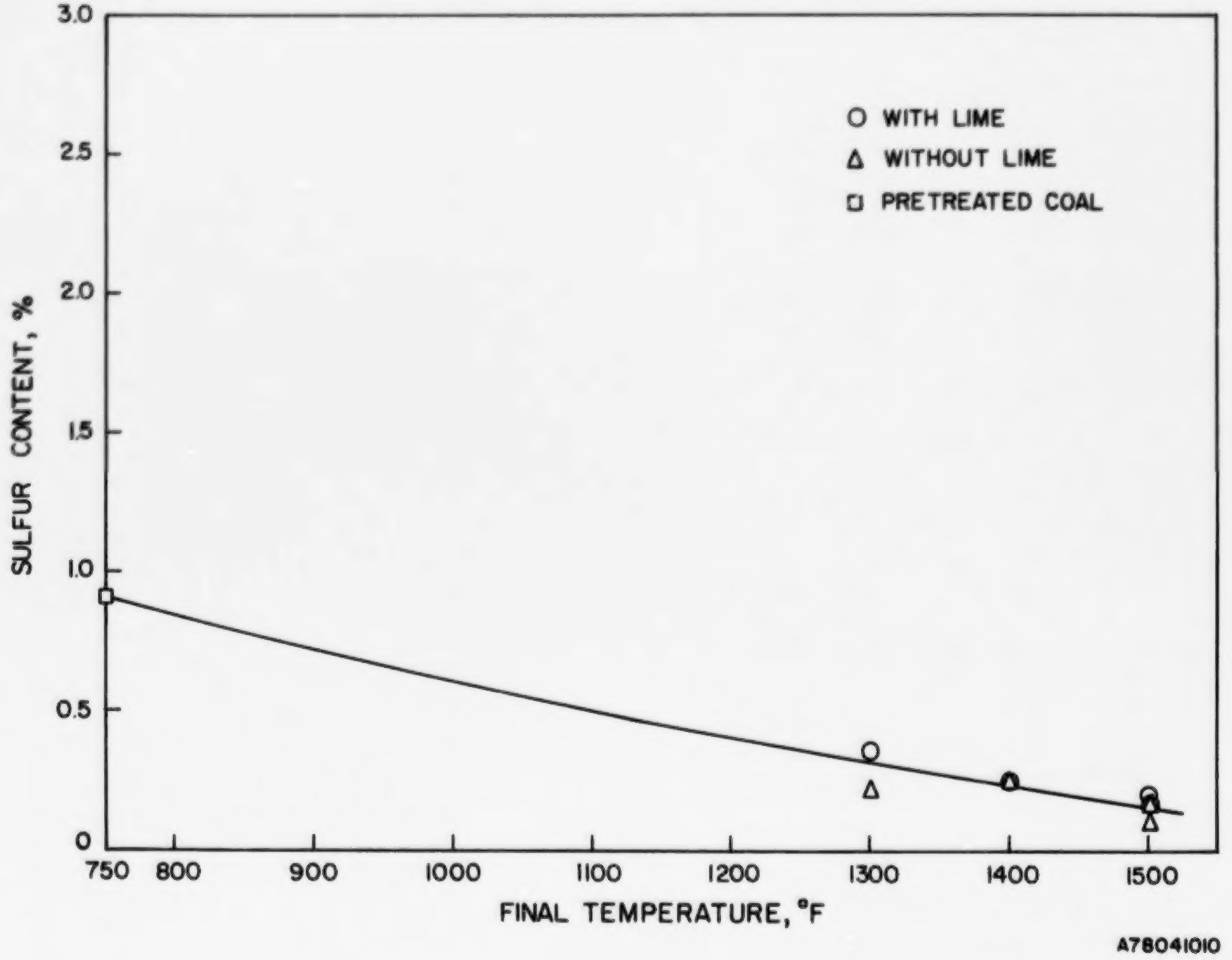

Figure 8. Treated material sulfur content, Pittsburgh seam, Pennsylvania coal.

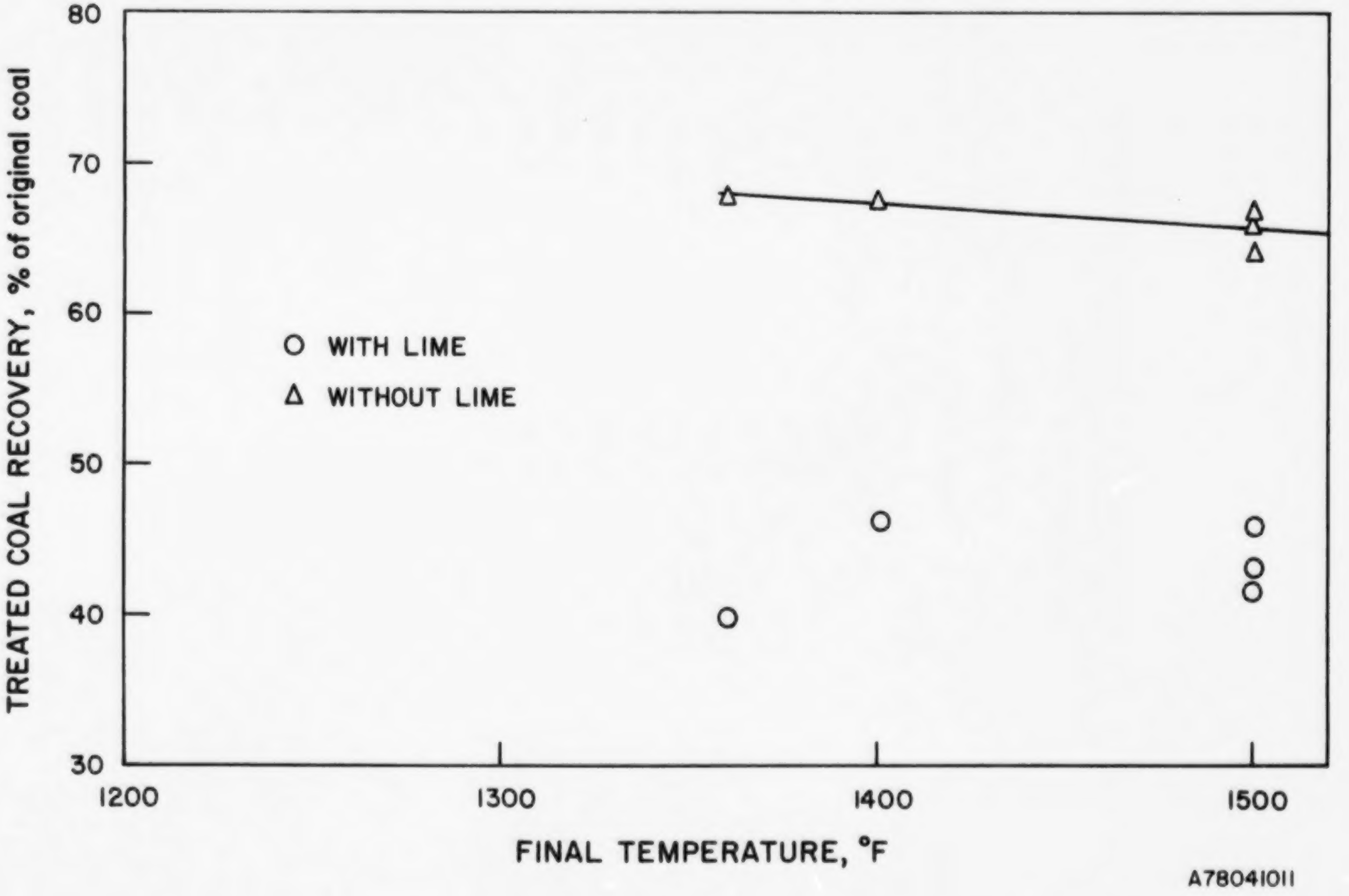

Figure 9. Treated material recovery, Pittsburgh seam, Pennsylvania coal.

TABLE 17. SO_2 EMISSIONS FOR THERMOBALANCE RUNS, PRETREATED PITTSBURGH SEAM COAL (PENNSYLVANIA MINE)

	$SO_2/10^6$ Btu	
Test No.	Total Sulfur	Pyritic and Organic Sulfur Only
TB-76-25	0.83	0.74
TB-76-26	0.83	0.48
TB-76-27	0.79	0.57
TB-76-28	0.76	0.55
TB-76-29	0.59	0.44
TB-76-30	0.83	0.35
TB-76-31	0.59	0.39
TB-76-32	0.72	0.26
TB-76-33	0.46	0.33
TB-76-34	0.74	0.28
Pretreated Coal	1.99	1.24
Raw Coal	2.21	1.22

TABLE 18. ILLINOIS NO. 6 COAL ANALYSIS

	Coal	Pretreated Coal	+40 Mesh Pretreated Coal
	wt %		
Proximate Analysis			
Moisture	1.3	0.4	1.6
Volatile Matter	37.3	28.5	27.6
Ash	8.5	9.4	8.8
Fixed Carbon	52.9	61.7	62.0
Total	100.0	100.0	100.0
Ultimate Analysis (dry)			
Ash	8.63	9.42	8.98
Carbon	74.50	75.20	74.80
Hydrogen	4.91	4.30	4.29
Sulfur	2.77	2.16	2.34
Nitrogen	1.49	1.66	1.55
Oxygen	7.70	7.26	8.04
Total	100.00	100.00	100.00

TABLE 19. THERMOBALANCE RUN DATA, ILLINOIS NO. 6 COAL

Run No.	Feed Coal*	Pretreated Coal*	TB-76-35			TB-76-36	
Feed Ratio Lime/Coal			2:1			0:1	
Heating Rate, °F/min			5			5	
Terminal Temperature, °F		750	1300			1300	
Holding Time, min		30	30			30	
Sulfur, wt %			Feed	Float	Sink	Feed	Residue
Sulfide	0.00	0.01	0.00	0.04	0.39	0.01	0.05
Sulfate	0.01	0.01	0.00	0.00	0.06	0.01	0.00
Pyritic	0.99	0.63	0.21	0.02	0.00	0.63	0.00
Organic	1.28	1.39	0.47	0.39	0.04	1.39	0.41
Total	2.28	2.04	0.68	0.45	0.49	2.04	0.46
Weight, g							
Initial	100		4.3094			2.4850	
Treated		90.4					1.7840
Weight Loss, %							
Total Weight		9.6		12.26			28.21
Coal Weight		9.6		36.79			28.21
Reduced Data							
Weight, lb	100.00		271.20	63.79	174.16	90.4	64.90
Sulfur Weight, lb							
Sulfide	0.00	0.01	0.01	0.03	0.68	0.01	0.03
Sulfate	0.01	0.01	0.01	0.00	0.10	0.01	0.00
Pyritic	0.99	0.57	0.57	0.01	0.00	0.57	0.00
Organic	1.28	1.26	1.26	0.25	0.07	1.26	0.27
Total	2.28	1.85	1.85	0.29	0.85	1.85	0.30
Sulfur Content, %							
Sulfide	0.00	0.01	0.00			0.01	
Sulfate	0.01	0.01	0.00			0.01	
Pyritic	0.99	0.63	0.21	0.02		0.63	0.00
Organic	1.28	1.39	0.47	0.39		1.39	0.41
Total	2.28	2.04	0.68	0.41		2.04	0.41
Sulfur Removal, wt %							
From Feed				85.9			85.4
From Coal		18.9		88.6			88.2

*Calculated for +40 mesh fraction.

TABLE 19. THERMOBALANCE RUN DATA, ILLINOIS NO. 6 COAL (Continued)

Run No.	Feed Coal*	Pretreated Coal*	TB-76-37			TB-76-38	
Feed Ratio Lime/Coal				2:1		0:1	
Heating Rate, °F/min				5		5	
Terminal Temperature, °F		750		1400		1400	
Holding Time, min		30		30		30	
Sulfur, wt %			Feed	Float	Sink	Feed	Residue
Sulfide	0.00	0.01	0.00	0.03	0.43	0.01	0.04
Sulfate	0.01	0.01	0.00	0.01	0.07	0.01	0.00
Pyritic	0.99	0.63	0.21	0.02	0.00	0.63	0.00
Organic	1.28	1.39	0.47	0.32	0.05	1.39	0.40
Total	2.28	2.04	0.68	0.38	0.55	2.04	0.44
Weight, g							
Initial	100		4.3980			2.6045	
Treated		90.4					1.8330
Weight Loss, %							
Total Weight		9.6		12.52			29.62
Coal Weight		9.6		37.57			29.62
Reduced Data							
Weight, lb	100.00		271.20	63.49	173.75	90.4	63.62
Sulfur Weight, lb							
Sulfide	0.00	0.01	0.01	0.02	0.75	0.01	0.03
Sulfate	0.01	0.01	0.01	0.01	0.12	0.01	0.00
Pyritic	0.99	0.57	0.57	0.01	0.00	0.57	0.00
Organic	1.28	1.26	1.26	0.20	0.09	1.26	0.25
Total	2.28	1.85	1.85	0.24	0.96	1.85	0.28
Sulfur Content, %							
Sulfide	0.00	0.01	0.00			0.01	
Sulfate	0.01	0.01	0.00			0.01	
Pyritic	0.99	0.63	0.21	0.02		0.63	0.00
Organic	1.28	1.39	0.47	0.32		1.39	0.40
Total	2.28	2.04	0.68	0.34		2.04	0.40
Sulfur Removal, wt %							
From Feed				88.6			86.5
From Coal		18.9		90.8			89.0

*Calculated for +40 mesh fraction.

TABLE 19. THERMOBALANCE RUN DATA, ILLINOIS NO. 6 COAL (Continued)

Run No.	Feed Coal*	Pretreated Coal*	TB-76-39			TB-76-40	
Feed Ratio Lime/Coal			2:1			0:1	
Heating Rate, °F/min			5			5	
Terminal Temperature, °F		750	1500			1500	
Holding Time, min		30	0			0	
			Feed	Float	Sink	Feed	Residue
Sulfur, wt %							
Sulfide	0.00	0.01	0.00	0.05	0.13	0.01	0.04
Sulfate	0.01	0.01	0.00	0.05	0.12	0.01	0.00
Pyritic	0.99	0.63	0.21	0.02	0.00	0.63	0.02
Organic	1.28	1.39	0.47	0.34	0.07	1.39	0.41
Total	2.28	2.04	0.68	0.46	0.32	2.04	0.47
Weight, g							
Initial	100		4.4136			2.5319	
Treated		90.4		1.0251	2.8346		1.7761
Weight Loss, %							
Total Weight		9.6		12.55			29.85
Coal Weight		9.6		37.64			29.85
Reduced Data							
Weight, lb	100.00		271.20	62.99	174.17	90.4	63.42
Sulfur Weight, lb							
Sulfide	0.00	0.01	0.01	0.03	0.23	0.01	0.03
Sulfate	0.01	0.01	0.01	0.03	0.21	0.01	0.00
Pyritic	0.99	0.57	0.57	0.01	0.00	0.57	0.01
Organic	1.28	1.26	1.26	0.21	0.12	1.26	0.26
Total	2.28	1.85	1.85	0.30	0.56	1.85	0.30
Sulfur Content, %							
Sulfide	0.00	0.01	0.00			0.01	
Sulfate	0.01	0.01	0.00			0.01	
Pyritic	0.99	0.63	0.21	0.02		0.63	0.02
Organic	1.28	1.39	0.47	0.34		1.39	0.41
Total	2.28	2.04	0.68	0.36		2.04	0.43
Sulfur Removal, wt %							
From Feed				88.1			85.4
From Coal		18.9		90.4			88.2

*Calculated for +40 mesh fraction.

TABLE 19. THERMOBALANCE RUN DATA, ILLINOIS NO. 6 COAL (Continued)

Run No.	Feed Coal*	Pretreated Coal*	TB-76-41			TB-76-42	
Feed Ratio Lime/Coal			2:1			0:1	
Heating Rate, °F/min			5			5	
Terminal Temperature, °F		750	1500			1500	
Holding Time, min		30	30			30	
Sulfur, wt %			Feed	Float	Sink	Feed	Residue
Sulfide	0.00	0.01	0.00	0.06	0.13	0.01	0.04
Sulfate	0.01	0.01	0.00	0.03	0.14	0.01	0.00
Pyritic	0.99	0.63	0.21	0.02	0.00	0.63	0.03
Organic	1.28	1.39	0.47	0.34	0.02	1.39	0.37
Total	2.28	2.04	0.68	0.45	0.29	2.04	0.44
Weight, g							
Initial	100		4.3986			2.4049	
Treated		90.4					1.6678
Weight Loss, %							
Total Weight		9.6		13.04			30.65
Coal Weight		9.6		39.11			30.65
Reduced Data							
Weight, lb	100.00		271.20	55.05	180.79	90.4	62.69
Sulfur Weight, lb							
Sulfide	0.00	0.01	0.01	0.03	0.24	0.01	0.03
Sulfate	0.01	0.01	0.01	0.02	0.25	0.01	0.00
Pyritic	0.99	0.57	0.57	0.01	0.00	0.57	0.02
Organic	1.28	1.26	1.26	0.19	0.04	1.26	0.23
Total	2.28	1.85	1.85	0.25	0.53	1.85	0.28
Sulfur Content, %							
Sulfide	0.00	0.01	0.00			0.01	
Sulfate	0.01	0.01	0.00			0.01	
Pyritic	0.99	0.63	0.21	0.02		0.63	0.03
Organic	1.28	1.39	0.47	0.34		1.39	0.37
Total	2.28	2.04	0.68	0.36		2.04	0.40
Sulfur Removal, wt %							
From Feed				89.2			86.5
From Coal		18.9		91.2			89.0

*Calculated for +40 mesh fraction.

TABLE 19. THERMOBALANCE RUN DATA, ILLINOIS NO. 6 COAL (Continued)

Run No.	Feed Coal*	Pretreated Coal*	TB-76-43			TB-76-44	
Feed Ratio Lime/Coal			2:1			0:1	
Heating Rate, ^{o}F/min			20			20	
Terminal Temperature, ^{o}F		750	1500			1500	
Holding Time, min		30	240			240	
			Feed	Float	Sink	Feed	Residue
Sulfur, wt %							
Sulfide	0.00	0.01	0.00	0.04	0.10	0.01	0.07
Sulfate	0.01	0.01	0.00	0.00	0.13	0.01	0.00
Pyritic	0.99	0.63	0.21	0.03	0.00	0.63	0.03
Organic	1.28	1.39	0.47	0.24	0.06	1.39	0.26
Total	2.28	2.04	0.68	0.31	0.29	2.04	0.36
Weight, g							
Initial	100		4.2878			2.3421	
Treated		90.4					1.6189
Weight Loss, %							
Total Weight		9.6		14.08			30.88
Coal Weight		9.6		42.24			30.88
Reduced Data							
Weight, lb	100.00		271.20	53.52	179.49	90.84	62.78
Sulfur Weight, lb							
Sulfide	0.00	0.01	0.01	0.02	0.18	0.01	0.04
Sulfate	0.01	0.01	0.01	0.00	0.23	0.01	0.00
Pyritic	0.99	0.57	0.57	0.02	0.00	0.57	0.02
Organic	1.28	1.26	1.26	0.13	0.11	1.26	0.16
Total	2.28	1.85	1.85	0.17	0.52	1.85	0.22
Sulfur Content, %							
Sulfide	0.00	0.01	0.00			0.01	
Sulfate	0.01	0.01	0.00			0.01	
Pyritic	0.99	0.63	0.21	0.03		0.63	0.03
Organic	1.28	1.39	0.47	0.24		1.39	0.26
Total	2.28	2.04	0.68	0.27		2.04	0.29
Sulfur Removal, wt %							
From Feed				91.9			90.3
From Coal		18.9		93.4			92.1

*Calculated for +40 mesh fraction.

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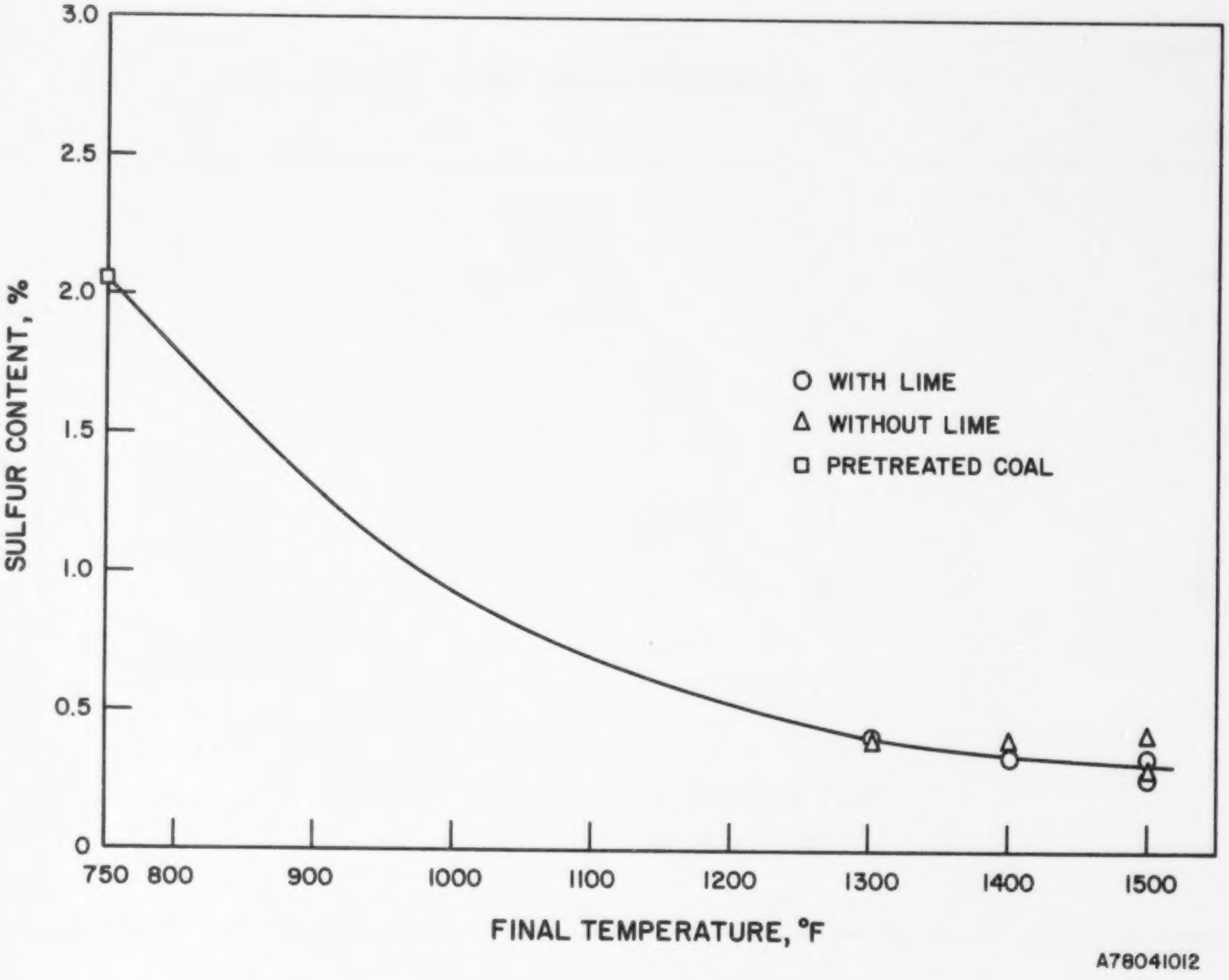

Figure 10. Treated material sulfur content, Illinois No. 6 coal.

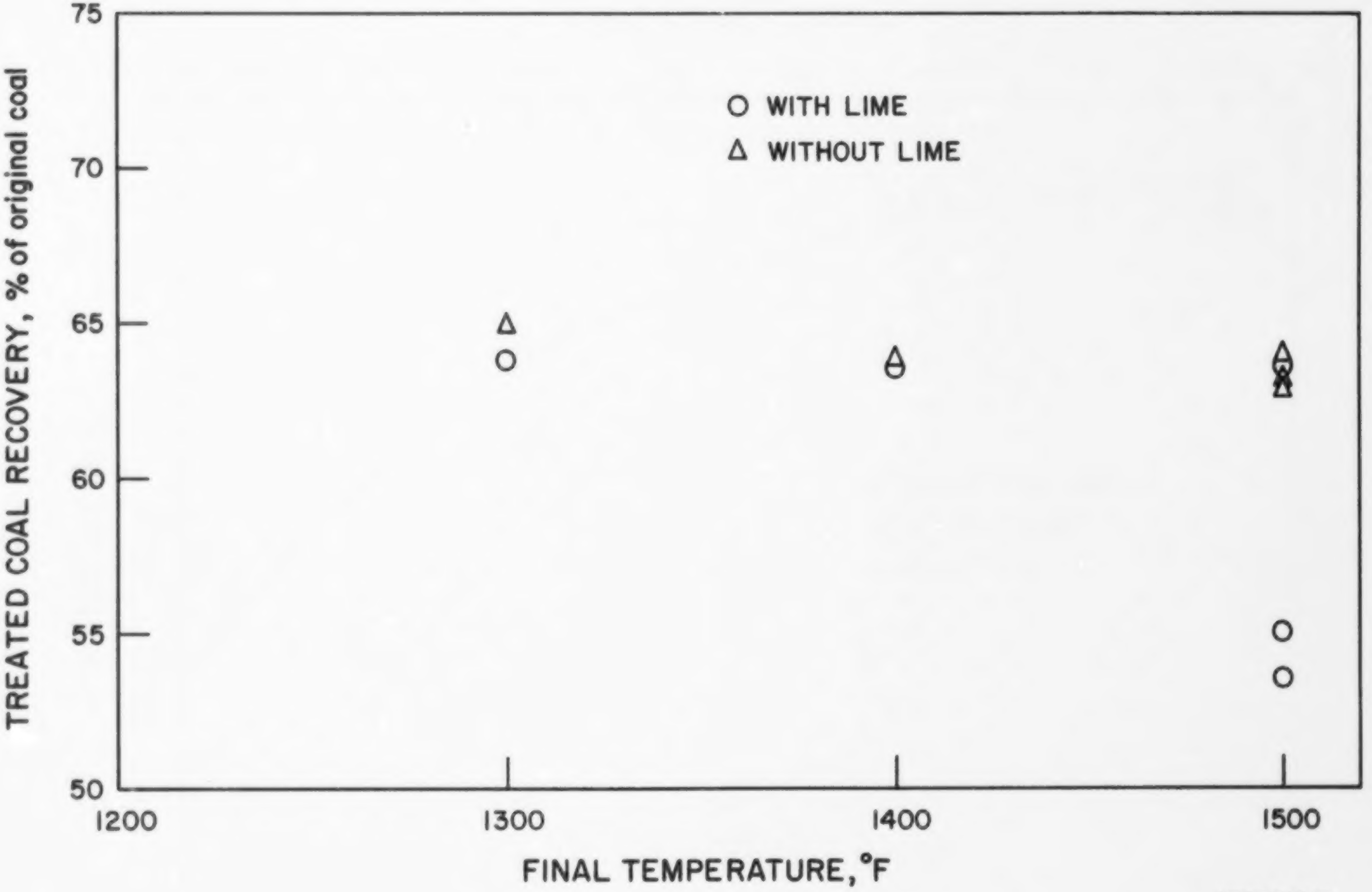

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Figure 11. Treated material recovery, Illinois No. 6 coal.

TABLE 20. SO_2 EMISSIONS FOR THERMOBALANCE RUNS, PRETREATED ILLINOIS NO. 6 COAL

	lb $SO_2/10^6$ Btu	
Test No.	Total Sulfur	Pyritic and Organic Sulfur Only
TB-76-35	0.72	0.66
TB-76-36	0.74	0.66
TB-76-37	0.61	0.54
TB-76-38	0.70	0.64
TB-76-39	0.74	0.58
TB-76-40	0.75	0.69
TB-76-41	0.72	0.58
TB-76-42	0.70	0.64
TB-76-43	0.50	0.43
TB-76-44	0.58	0.46
Pretreated Coal	3.13	3.10
Raw Coal	3.41	3.39

EFFECT OF HEAT-UP RATE

Some rapid heat-up tests were made with pretreated Western Kentucky No. 9 coal in the thermobalance. Effects of rapid heating rate and prolonged residence times on sulfur removal, with and without lime, were studied. The rapid heat-up is accomplished by lowering the basket into the zone that is at 1500°F, rather than lowering it and then applying heat.

These runs (TB-76-21 through TB-76-24, Table 21) all show good sulfur reduction and nearly the same weight loss. The recovery is higher for treated material in the no-lime tests.

A graph of the sample weight loss as it is heated to 1500°F is shown in Figure 12. The curve is divided into three sections: A, B, and C. Section A is the weight loss observed while the sample is in the cool zone. This loss is caused by convection heating from the hot zone below the basket; the weight loss is the moisture removed in this time period. Section B is the weight loss as the sample is lowered into the heated zone; section C is the weight loss during the holding time. Although the curves differ, the final point is essentially the same regardless of the heating rate.

Pretreated Western Kentucky No. 9 coal without lime was used in additional thermobalance tests with rapid heat-up to determine the effect of shortened soaking time. The samples were rapidly heated to 1500°F and soaked at that temperature for 15, 30, 60, and 90 minutes.

Figure 13 shows the weight loss with respect to residence time in the heated zone. The initial broken line is attributed to weight loss by moisture reduction as the unit is heated up. When the heated zone reaches 1500°F, the sample basket is lowered into it. The majority of the weight loss occurs in the first 24 seconds as the basket is lowered and positioned in the hot zone. Only slightly more weight loss is recorded at the end of 15 minutes and essentially none after this, up to 120 minutes.

The results for these tests, TB-76-45 through TB-76-48, are shown in Table 22. A previous run, TB-76-24, with rapid heat-up and 120-minute residence time is included for comparison. The tests show decreasing total sulfur with increased holding time at 1500°F. The pyritic sulfur content is reduced to nearly zero by the end of 15 minutes. The organic sulfur reduction is directly dependent upon residence time at temperature. Material recovery is reduced as the time increases.

Figure 14 illustrates the sulfur reduction achieved in the tests. As mentioned, the organic sulfur and total sulfur decrease as residence time increases. The SO_2 emissions for the tests are shown in Table 23. The heating value was estimated at 11,565 Btu/lb. A residence time of 60 minutes yields a borderline SO_2 emission, while longer times are acceptable, considering total sulfur. All tests are within limits if only pyritic and organic sulfur are used.

Based on these data, the coal can be heated rapidly to 1500°F, as by injection into a fluidized bed. A residence time of 15 to 60 minutes is sufficient for reduction of sulfur to acceptable emission limits.

TABLE 21. THERMOBALANCE RUN DATA, PRETREATED WESTERN KENTUCKY NO. 9 COAL

	Feed Coal	Pretreated Coal					
Run No.			TB-76-21			TB-76-22	
Feed Ratio, lime/coal			2:1			0:1	
Coal	W. Ky. No. 9		Pret. W. Ky. No. 9				
Heating Rate, °F/min.			20			20	
Terminal Temperature, °F		750	1500			1500	
Holding Time, min.		30	120			120	
			Feed	Float	Sink	Feed	Residue
Sulfur, wt %							
Sulfide	0.02	0.01	0.00	0.13	0.97	0.01	0.22
Sulfate	0.52	0.10	0.03	0.00	0.03	0.10	0.01
Pyritic	0.92	1.25	0.42	0.03	0.00	1.25	0.01
Organic	1.57	1.29	0.43	0.29	0.05	1.29	0.19
Total	3.03	2.65	0.88	0.45	1.05	2.65	0.43
Weight, gm							
Initial	100		4.0566			2.6063	
Treated		90.84		0.7146	2.7864		1.7699
Weight Loss, %							
Total Weight		9.16		13.70			32.09
Coal Weight		9.16		41.10			32.09
Reduced Data							
Weight, lb		90.84	272.52	48.00	187.18	90.84	61.69
Sulfur Weight, lb							
Sulfide	0.02	0.01	0.01	0.06	1.82	0.01	0.13
Sulfate	0.52	0.09	0.09	0.00	0.06	0.09	0.01
Pyritic	0.92	1.13	1.13	0.01	0.00	1.13	0.01
Organic	1.57	1.17	1.17	0.14	0.09	1.17	0.12
Total	3.03	2.40	2.40	0.22	1.97	2.40	0.27
Sulfur Content, %							
Sulfide	0.02	0.01	0.00			0.01	
Sulfate	0.52	0.10	0.03			0.10	
Pyritic	0.92	1.25	0.42	0.03		1.25	0.01
Organic	1.57	1.29	0.43	0.29		1.29	0.19
Total	3.03	2.65	0.88	0.32		2.65	0.20
Sulfur Removal, wt %							
From Feed				93.75			94.58
From Coal		20.5		93.05			95.71

*Sample was lowered rapidly into reaction zone at 1500°F.

TABLE 21. THERMOBALANCE RUN DATA, PRETREATED WESTERN KENTUCKY NO. 9 COAL (Continued)

	Feed Coal	Pretreated Coal					
Run No.			TB-76-23			TB-76-24	
Feed Ratio, lime/coal			2:1			0:1	
Coal	W. Ky. No. 9		Pret. W. Ky. No. 9				
Heating Rate, °F/min.			Rapid Heating*			Rapid Heating*	
Terminal Temperature, °F		750	1500			1500	
Holding Time, min.		30	120			120	
			Feed	Float	Sink	Feed	Residue
Sulfur, wt %							
Sulfide	0.02	0.01	0.00	0.13	1.09	0.01	0.13
Sulfate	0.52	0.10	0.03	0.00	0.03	0.10	0.00
Pyritic	0.92	1.25	0.42	0.03	0.00	1.25	0.02
Organic	1.57	1.29	0.43	0.16	0.05	1.29	0.14
Total	3.03	2.65	0.88	0.32	1.17	2.65	0.29
Weight, gm							
Initial	100		4.2964			2.5892	
Treated		90.84		0.8022	2.9100		1.7646
Weight Loss, %							
Total Weight		9.16		13.60			31.85
Coal Weight		9.16		40.79			31.85
Reduced Data							
Weight, lb		90.84	272.52	50.88	184.57	90.84	61.91
Sulfur Weight, lb							
Sulfide	0.02	0.01	0.01	0.07	2.01	0.01	0.08
Sulfate	0.52	0.09	0.09	0.00	0.06	0.09	0.00
Pyritic	0.92	1.13	1.13	0.01	0.00	1.13	0.01
Organic	1.57	1.17	1.17	0.08	0.09	1.17	0.09
Total	3.03	2.40	2.40	0.16	2.16	2.40	0.18
Sulfur Content, %							
Sulfide	0.02	0.01	0.00			0.01	
Sulfate	0.52	0.10	0.03			0.10	
Pyritic	0.92	1.25	0.42	0.03		1.25	0.02
Organic	1.57	1.29	0.43	0.16		1.29	0.14
Total	3.03	2.65	0.88	0.19		2.65	0.16
Sulfur Removal, wt %							
From Feed				96.25			95.83
From Coal		20.5		97.03			96.70

*Sample was lowered rapidly into reaction zone at 1500°F.

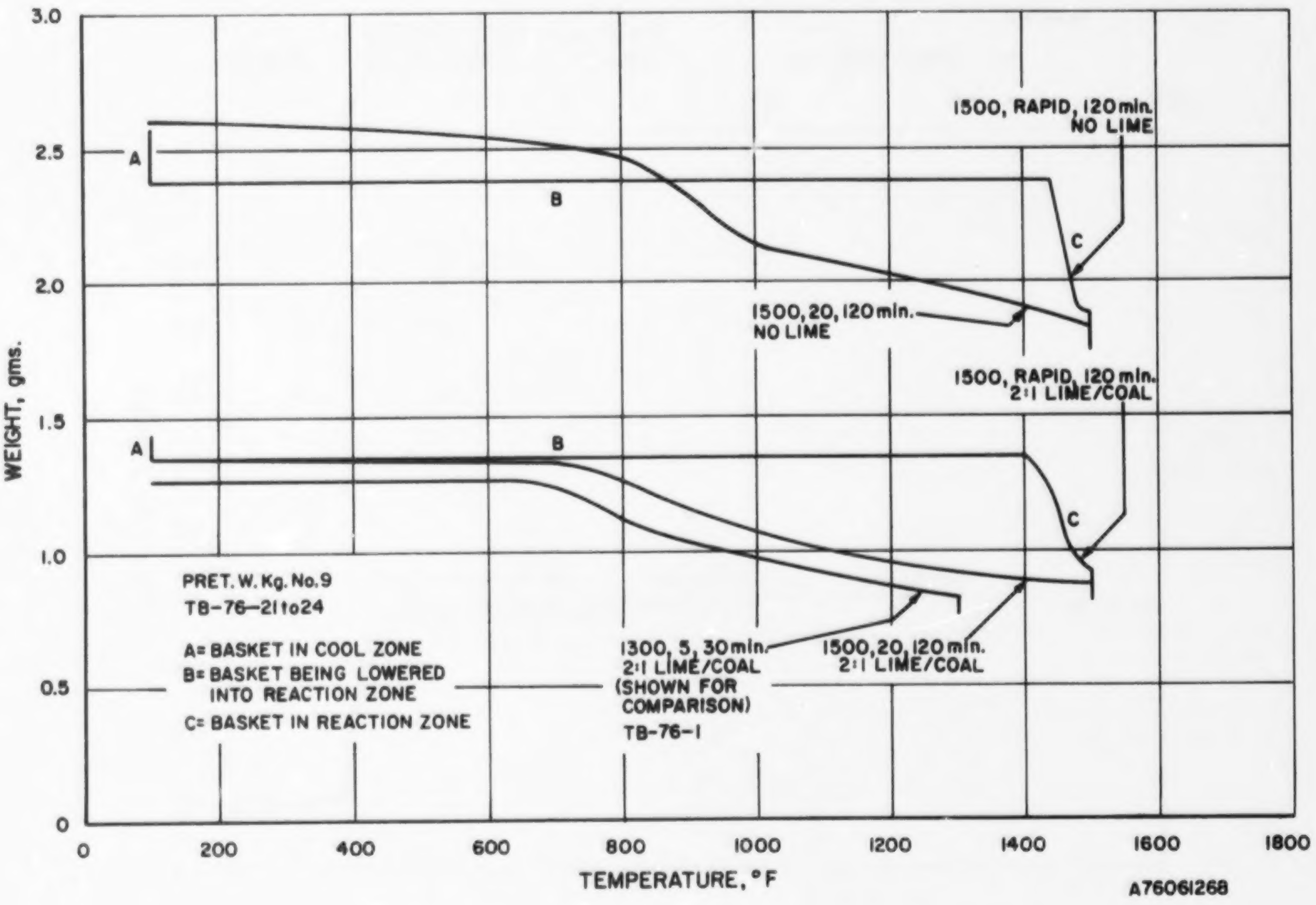

Figure 12. Thermobalance sample weight loss.

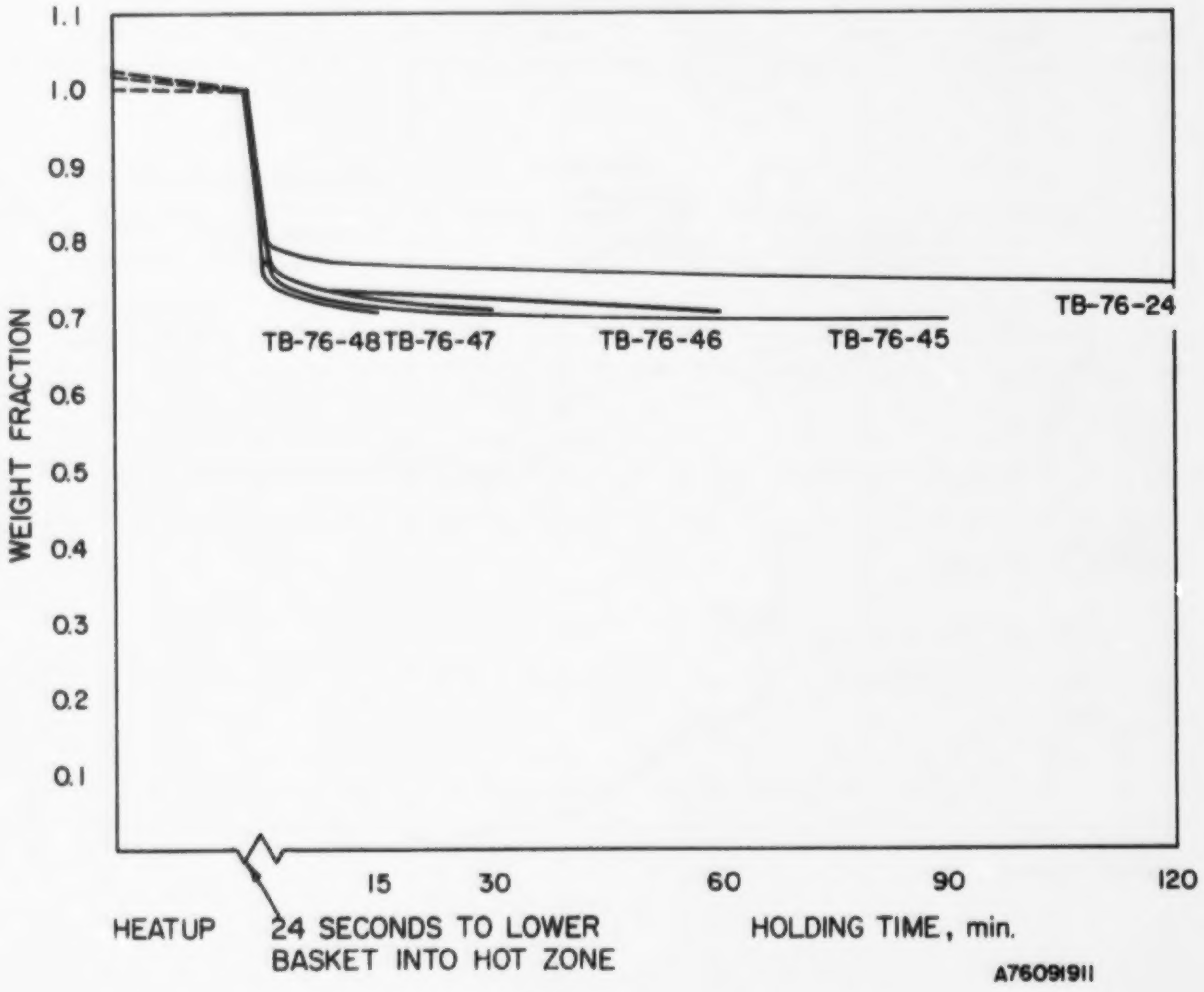

Figure 13. Thermobalance runs, data on pretreated Western Kentucky No. 9 coal.

TABLE 22. THERMOBALANCE RUN DATA, WESTERN KENTUCKY NO. 9 COAL (RAPID HEAT-UP RATE)

Run No.	Feed Coal*	Pretreated Coal*	TB-76-24		TB-76-45		TB-76-46	
Lime/Coal Feed Ratio			0:1		0:1		0:1	
Coal	W. Ky. No. 9		Pretreated W. Ky. No. 9					
Heating Rate, °F/min			Rapid Heating		Rapid Heating		Rapid Heating	
Terminal Temperature, °F		750	1500		1500		1500	
Holding Time, min		30	120		90		60	
			Feed	Residue	Feed	Residue	Feed	Residue
Sulfur, wt %								
Sulfide	0.02	0.01	0.01	0.13	0.01	0.23	0.01	0.23
Sulfate	0.52	0.10	0.10	0.00	0.10	0.00	0.10	0.01
Pyritic	0.92	1.25	1.25	0.02	1.25	0.02	1.25	0.02
Organic	1.57	1.29	1.29	0.14	1.29	0.32	1.29	0.46
Total	3.03	2.65	2.65	0.29	2.65	0.57	2.65	0.72
Weight, g								
Initial	100		2.5892		2.9610		2.8700	
Treated		90.84		1.7646		2.0686		2.0121
Weight Loss, %								
Total Weight		9.16		31.85		30.14		29.89
Coal Weight		9.16		31.85		30.14		29.89
Reduced Data								
Weight, lb		90.84	90.84	61.91	90.84	63.46	90.84	63.69
Sulfur Weight, lb								
Sulfide	0.02	0.01	0.01	0.08	0.01	0.15	0.01	0.15
Sulfate	0.52	0.09	0.09	0.00	0.09	0.00	0.09	0.01
Pyritic	0.92	1.13	1.13	0.01	1.13	0.01	1.13	0.01
Organic	1.57	1.17	1.17	0.09	1.17	0.20	1.17	0.29
Total	3.03	2.40	2.40	0.18	2.40	0.36	2.40	0.46
Sulfur Content, %								
Sulfide	0.02	0.01	0.01		0.01		0.01	
Sulfate	0.52	0.10	0.10		0.10		0.10	
Pyritic	0.92	1.25	1.25	0.02	1.25	0.02	1.25	0.02
Organic	1.57	1.29	1.29	0.14	1.29	0.32	1.29	0.46
Total	3.03	2.65	2.65	0.16	2.65	0.34	2.65	0.48
Sulfur Removal, wt %								
From Feed				95.8		91.3		87.5
From Coal		20.5		96.7		93.1		90.1

*Calculated for +40 mesh.

TABLE 22. THERMOBALANCE RUN DATA, WESTERN KENTUCKY NO. 9 COAL (RAPID HEAT-UP RATE) (Continued)

Run No.	Feed Coal*	Pretreated Coal*	TB-76-47		TB-76-48	
Lime/Coal Feed Ratio			0:1		0:1	
Coal	W. Ky. No. 9		Pretreated W. Ky. No. 9			
Heating Rate, °F/min			Rapid Heating		Rapid Heating	
Terminal Temperature, °F		750	1500		1500	
Holding Time, min		30	30		15	
			Feed	Residue	Feed	Residue
Sulfur, wt %						
Sulfide	0.02	0.01	0.01	0.41	0.01	0.50
Sulfate	0.52	0.10	0.10	0.01	0.10	0.02
Pyritic	0.92	1.25	1.25	0.02	1.25	0.00
Organic	1.57	1.29	1.29	0.57	1.29	0.68
Total	3.03	2.65	2.65	1.01	2.65	1.20
Weight, g						
Initial	100		2.7170		2.5688	
Treated		90.84		1.9329		1.8493
Weight Loss, %						
Total Weight		9.16		28.86		28.00
Coal Weight		9.16		28.86		28.00
Reduced Data						
Weight, lb		90.84	90.84	64.62	90.84	65.40
Sulfur Weight, lb						
Sulfide	0.02	0.01	0.01	0.26	0.01	0.33
Sulfate	0.52	0.09	0.09	0.01	0.09	0.01
Pyritic	0.92	1.13	1.13	0.01	1.13	0.00
Organic	1.57	1.17	1.17	0.37	1.17	0.44
Total	3.03	2.40	2.40	0.65	2.40	0.78
Sulfur Content, %						
Sulfide	0.02	0.01	0.01		0.01	
Sulfate	0.52	0.10	0.10		0.10	
Pyritic	0.92	1.25	1.25	0.02	1.25	0.00
Organic	1.57	1.29	1.29	0.57	1.29	0.68
Total	3.03	2.65	2.65	0.59	2.65	0.68
Sulfur Removal, wt %						
From Feed				84.2		81.7
From Coal		20.5		87.5		85.5

B76092055

*Calculated for +40 mesh.

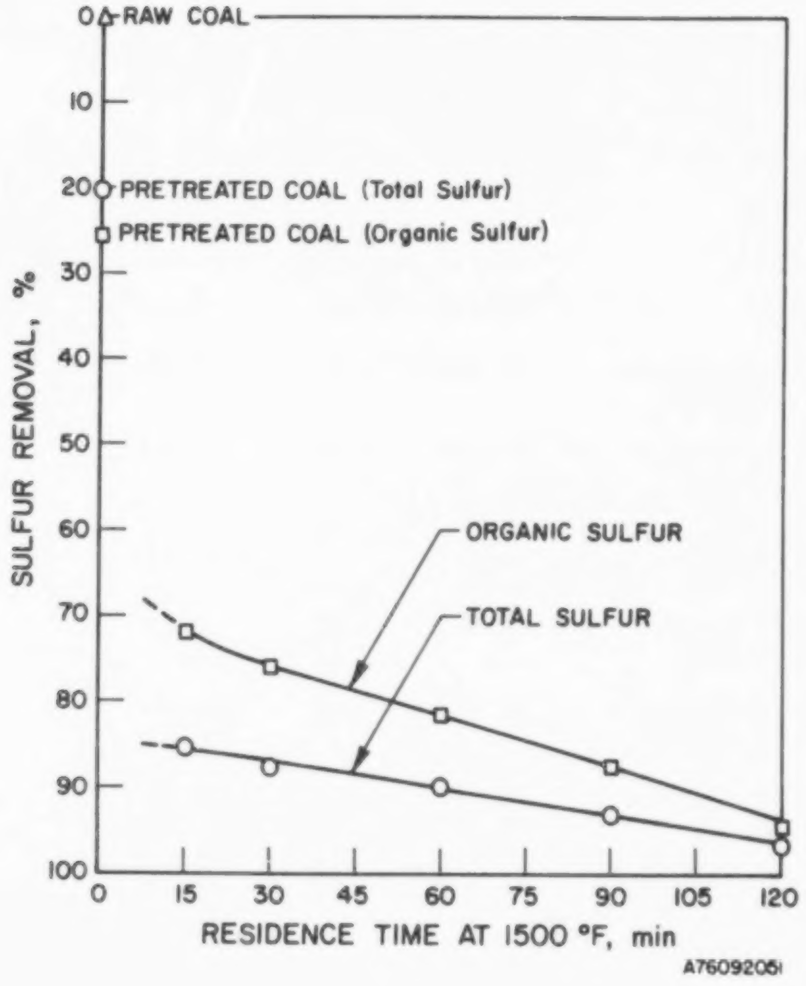

Figure 14. Effect of holding time on sulfur removal.

TABLE 23. SO_2 EMISSIONS AND HEATING VALUES FOR WESTERN KENTUCKY NO. 9 COAL (RAPID HEAT-UP RATE)

Treatment Conditions (Test No.)	Btu/lb	lb $SO_2/10^6$ Btu Total	lb $SO_2/10^6$ Btu Pyritic and Organic	Total Heating Value, Btu/100 lb Coal Feed
Coal (dry)	12,454	5.62	4.63	1,245,400
Pretreated +40 mesh 750°F, 30 min	11,809	5.18	4.49	1,072,730
1500°F, 15 min (TB-76-48)	11,565	2.07	1.17	756,351
1500°F, 30 min (TB-76-47)	11,565	1.74	1.02	747,330
1500°F, 60 min (TB-76-46)	11,565	1.24	0.83	736,575
1500°F, 90 min (TB-76-45)	11,565	0.98	0.59	733,915
1500°F, 120 min (TB-76-24)	11,565	0.50	0.28	715,989
1500°F, 330 min (BR-76-7)	11,565	0.66	0.28	717,146

Note: EPA limit for SO_2 emissions = 1.2 lb $SO_2/10^6$ Btu.

LABORATORY-SCALE TEST CONCLUSIONS

The data obtained from the thermobalance reactor support the following conclusions:

- *Four Eastern U.S. coals have been satisfactorily hydrodesulfurized. These coals included run-of-mine coal, high-ash coal, high-sulfur coal, and both mid-continent and Appalachian coals.*
- *Pretreatment of the coals is required for satisfactory sulfur removal.*
- *The use of the lime sulfur-getter is not imperative; coal recovery is greater if lime is not used.*
- *Rapid heat-up is satisfactory for sulfur removal.*
- *Temperatures of 1400° to 1500°F are desirable with a total reaction time of over 60 minutes.*

BENCH-SCALE TEST WORK – HYDRODESULFURIZATION

WESTERN KENTUCKY NO. 9 COAL RUNS

Slow Heat-Up Runs

Data for test runs with slow heat-up rate with pretreated Western Kentucky No. 9 coal are shown in Tables 24 through 26. The pretreated coal was screened at +40 mesh so that the size consist would be the same as in the thermobalance runs. The lime used was −60+80 mesh for compatible fluidization characteristics and so that a screen separation of treated material could be made at +50 mesh. Analyses for the coal, pretreated coal, and +40 mesh pretreated coal are shown in Table 24.

Table 25 lists laboratory data and reduced data. Values for coal and pretreated coal have been back-calculated to show only +40 mesh materials because of the uneven sulfur distribution. The reduced data for the tests using lime were calculated for both screen and float-sink separation. Treated material was split into two parts by riffling. One part was screen separated and the other float-sink separated. To put all data on the same basis, the fractions were treated as if they were from the entire sample. For example, assume 150 grams of mixture were initially charged, and 125 grams remained after treatment. This material is split into two parts by riffling. One part is separated by 50 mesh screening and the percentages of +50 and −50 mesh material recorded. These percentages are used to determine how the total 125 grams would have separated and the reduced data calculated accordingly. The same procedure is used for the float-sink separation results in calculations. In this way, each lime/coal mixture run yielded not only a comparison with the no-lime runs, but the separation techniques as well.

Identical conditions were used in each pair of experiments, one with coal only and the other with the lime/coal mix. Heating rate, terminal temperatures, and holding times were varied as shown in Table 25. Comparison of the pairs shows that the no-lime tests have higher total sulfur values (less complete removal) than the lime tests. Part of this can be attributed to the removal of the sulfide-type sulfur in the separation processes. The reduced data on pyritic and organic sulfur removal do not exhibit as large a difference. Treated material recovery is higher in all the coal-only tests than either of the separation techniques for the lime test.

Contrary to the thermobalance data for this coal, reported earlier, sulfur removal appears to be better in tests using the coal/lime mixture. The reason may be in more intimate contact and mixing in the fluidized condition of the batch reactor, as compared with the thermobalance stagnant material conditions. Other factors, such as amount of material handled, gas requirements for treatment of the lime and coal mix, and the separation facilities, favor operation without lime.

Table 26 gives the calculated SO_2 emissions and product heat recovery from the treated coal. On this basis, only the first pair of runs shows a large difference in SO_2

TABLE 24. ANALYSIS OF BATCH REACTOR RUNS WITH WESTERN KENTUCKY NO. 9 COAL

	Coal	Pretreated Coal	+40 Mesh Pretreated Coal
	wt %		
Proximate Analysis			
Moisture	5.8	0.8	1.6
Volatile Matter	36.3	27.7	26.7
Ash	10.6	11.2	14.1
Fixed Carbon	47.3	60.3	57.6
Total	100.0	100.0	100.0
Ultimate Analysis			
Ash	11.24	11.25	14.31
Carbon	70.00	71.40	67.80
Hydrogen	4.54	4.06	3.84
Sulfur	3.74	3.16	3.23
Nitrogen	1.53	1.64	1.48
Oxygen	8.95	8.49	9.34
Total	100.00	100.00	100.00

TABLE 25. BATCH REACTOR TEST RUN DATA FOR PRETREATED WESTERN KENTUCKY NO. 9 COAL

Run No.	Feed Coal*	+40 Mesh Pretreated Coal	BR-76-1		BR-76-2				
Lime/Coal Feed Ratio			0:1		2:1				
Coal	W. Ky. No. 9		Pretreated W. Ky. No. 9						
Heating Rate, °F/min			5		5				
Terminal Temperature, °F		750	1400		1400				
Holding Time, min		30	30		30				
			Feed	Residue	Feed	+50	−50	Float	Sink
Sulfur, wt %									
Sulfide	0.02	0.04	0.04	0.42	0.01	0.11	1.01	0.05	1.02
Sulfate	0.60	0.10	0.10	0.00	0.03	0.01	0.04	0.01	0.04
Pyritic	1.06	1.54	1.54	0.02	0.52	0.02	0.00	0.01	0.00
Organic	1.82	1.38	1.38	0.81	0.46	0.30	0.06	0.06	0.21
Total	3.50	3.06	3.06	1.25	1.02	0.44	1.11	0.13	1.27
Weight, g									
Initial	100.00		150		150				
Treated		90.84		109.8		126.4			
Weight Loss, %									
Total Weight		9.16		26.8		15.73			
Coal Weight		9.16		26.8		47.20			
Reduced Data									
Weight, lb			90.84	66.49	272.52	61.39	168.26	57.97	171.68
Sulfur Weight, lb									
Sulfide	0.02	0.04	0.04	0.30	0.04	0.07	1.70	0.03	1.75
Sulfate	0.60	0.09	0.09	0.00	0.09	0.01	0.07	0.01	0.07
Pyritic	1.06	1.40	1.40	0.01	1.40	0.01	0.00	0.01	0.00
Organic	1.82	1.25	1.25	0.54	1.25	0.18	0.10	0.03	0.36
Total	3.50	2.78	2.78	0.85	2.78	0.27	1.87	0.08	2.18
Sulfur Content %									
Sulfide	0.02	0.04	0.04		0.01				
Sulfate	0.60	0.10	0.10		0.03				
Pyritic	1.06	1.54	1.54	0.02	0.52	0.02		0.01	
Organic	1.82	1.38	1.38	0.81	0.46	0.30		0.06	
Total	3.50	3.06	3.06	0.83	1.02	0.32		0.07	
Sulfur Removal, wt %									
From Feed				80.2		93.2		98.6	
From Coal		20.6		84.3		94.6		98.9	

*Calculated for 40 mesh fraction.

TABLE 25. BATCH REACTOR TEST RUN DATA FOR PRETREATED WESTERN KENTUCKY NO. 9 COAL (Continued)

Run No.	Feed Coal*	+40 Mesh Pretreated Coal	BR-76-3		BR-76-4				
Lime/Coal Feed Ratio			0:1		2:1				
Coal	W. Ky. No. 9		Pretreated W. Ky. No. 9						
Heating Rate, °F/min			5		5				
Terminal Temperature, °F		750	1500		1500				
Holding Time, min		30	30		30				
			Feed	Residue	Feed	+50	−50	Float	Sink
Sulfur, wt %									
Sulfide	0.02	0.04	0.04	0.12	0.01	0.22	1.29	0.10	1.30
Sulfate	0.60	0.10	0.10	0.00	0.03	0.01	0.05	0.00	0.03
Pyritic	1.06	1.54	1.54	0.02	0.52	0.02	0.00	0.01	0.00
Organic	1.82	1.38	1.38	0.37	0.46	0.17	0.04	0.32	0.08
Total	3.50	3.06	3.06	0.51	1.02	0.42	1.38	0.43	1.41
Weight, g									
Initial	100.00		75.0		150				
Treated		90.84		51.7		121.9			
Weight Loss, %									
Total Weight		9.16		31.1		18.7			
Coal Weight		9.16		31.1		56.2			
Reduced Data									
Weight, lb			90.84	62.62	272.52	53.17	168.29	49.68	171.78
Sulfur Weight, lb									
Sulfide	0.02	0.04	0.04	0.08	0.04	0.12	2.17	0.05	2.23
Sulfate	0.60	0.09	0.09	0.00	0.09	0.01	0.08	0.00	0.05
Pyritic	1.06	1.40	1.40	0.01	1.40	0.01	0.00	0.01	0.00
Organic	1.82	1.25	1.25	0.23	1.25	0.09	0.07	0.16	0.14
Total	3.50	2.78	2.78	0.32	2.78	0.23	2.32	0.22	2.42
Sulfur Content %									
Sulfide	0.02	0.04	0.04		0.01				
Sulfate	0.60	0.10	0.10		0.03				
Pyritic	1.06	1.54	1.54	0.02	0.52	0.02		0.01	
Organic	1.82	1.38	1.38	0.37	0.46	0.17		0.32	
Total	3.50	3.06	3.06	0.39	1.02	0.19		0.33	
Sulfur Removal, wt %									
From Feed				91.4		96.4		93.9	
From Coal		20.6		93.1		97.1		95.1	

*Calculated for 40 mesh fraction.

TABLE 25. BATCH REACTOR TEST RUN DATA FOR PRETREATED WESTERN KENTUCKY NO. 9 COAL (Continued)

Run No.	Feed Coal*	+40 Mesh Pretreated Coal	BR-76-5		BR-76-6				
Lime/Coal Feed Ratio			0:1		2:1				
Coal	W. Ky. No. 9		Pretreated W. Ky. No. 9						
Heating Rate, °F/min			20		20				
Terminal Temperature, °F		750	1500		1500				
Holding Time, min		30	240		240				
			Feed	Residue	Feed	+50	−50	Float	Sink
Sulfur, wt %									
Sulfide	0.02	0.04	0.04	0.26	0.01	0.13	1.02	0.06	0.95
Sulfate	0.60	0.10	0.10	0.01	0.03	0.00	0.01	0.01	0.03
Pyritic	1.06	1.54	1.54	0.02	0.52	0.00	0.00	0.03	0.00
Organic	1.82	1.38	1.38	0.27	0.46	0.09	0.02	0.19	0.02
Total	3.50	3.06	3.06	0.56	1.02	0.22	1.05	0.29	1.00
Weight, g									
Initial	100.00		75		150				
Treated		90.84		41.3		119.4			
Weight Loss, %									
Total Weight		9.16		44.93		20.4			
Coal Weight		9.16		44.93		61.2			
Reduced Data									
Weight, lb			90.84	50.02	272.52	53.96	162.97	48.01	168.92
Sulfur Weight, lb									
Sulfide	0.02	0.04	0.04	0.13	0.04	0.07	1.66	0.03	1.60
Sulfate	0.60	0.09	0.09	0.01	0.09	0.00	0.02	0.00	0.05
Pyritic	1.06	1.40	1.40	0.01	1.40	0.00	0.00	0.01	0.00
Organic	1.82	1.25	1.25	0.14	1.25	0.05	0.03	0.09	0.03
Total	3.50	2.78	2.78	0.29	2.78	0.12	1.71	0.13	1.68
Sulfur Content %									
Sulfide	0.02	0.04	0.04	--	0.01	--		--	
Sulfate	0.60	0.10	0.10	--	0.03	--		--	
Pyritic	1.06	1.54	1.54	0.02	0.52	0.00		0.03	
Organic	1.82	1.38	1.38	0.27	0.46	0.09		0.19	
Total	3.50	3.06	3.06	0.29	1.02	0.09		0.22	
Sulfur Removal, wt %									
From Feed				94.6		98.2		96.4	
From Coal		20.6		95.7		98.6		97.1	

*Calculated for 40 mesh fraction.

TABLE 25. BATCH REACTOR TEST RUN DATA FOR PRETREATED WESTERN KENTUCKY NO. 9 COAL (Continued)

Run No.	Feed Coal*	+40 Mesh Pretreated Coal	BR-76-21		BR-76-22				
Lime/Coal Feed Ratio			0:1		2:1				
Coal	W. Ky. No. 9		Pretreated W. Ky. No. 9						
Heating Rate, °F/min			5		5				
Terminal Temperature, °F		750	1400		1400				
Holding Time, min		30	60		60				
			Feed	Residue	Feed	+50	−50	Float	Sink
Sulfur, wt %									
Sulfide	0.02	0.04	0.04	0.20	0.01	0.10	1.08	0.05	1.45
Sulfate	0.60	0.10	0.10	0.01	0.03	0.00	0.07	0.03	0.08
Pyritic	1.06	1.54	1.54	0.03	0.52	0.02	0.00	0.02	0.00
Organic	1.82	1.38	1.38	0.35	0.46	0.20	0.16	0.20	0.04
Total	3.50	3.06	3.06	0.59	1.02	0.32	1.31	0.30	1.57
Weight, g									
Initial	100.00		75.00		150				
Treated		90.84		52.5		119.8			
Weight Loss, %									
Total Weight		9.16		30.0		20.13			
Coal Weight		9.16		30.0		60.39			
Reduced Data									
Weight, lb			90.84	63.59	272.52	54.13	163.52	55.5	162.16
Sulfur Weight, lb									
Sulfide	0.02	0.04	0.04	0.13	0.04	0.05	1.77	0.03	2.35
Sulfate	0.60	0.09	0.09	0.01	0.09	0.00	0.11	0.02	0.13
Pyritic	1.06	1.40	1.40	0.02	1.40	0.01	0.00	0.01	0.00
Organic	1.82	1.25	1.25	0.22	1.25	0.11	0.26	0.11	0.06
Total	3.50	2.78	2.78	0.38	2.78	0.17	2.14	0.17	2.54
Sulfur Content %									
Sulfide	0.02	0.04	0.04	--	0.01	--		--	
Sulfate	0.60	0.10	0.10	--	0.03	--		--	
Pyritic	1.06	1.54	1.54	0.03	0.52	0.02		0.02	
Organic	1.82	1.38	1.38	0.35	0.46	0.20		0.20	
Total	3.50	3.06	3.06	0.38	1.02	0.22		0.22	
Sulfur Removal, wt %									
From Feed				91.4		95.7		95.7	
From Coal		20.6		93.1		96.6		96.6	

*Calculated for 40 mesh fraction.

TABLE 25. BATCH REACTOR TEST RUN DATA FOR PRETREATED WESTERN KENTUCKY NO. 9 COAL (Continued)

Run No.	Feed Coal*	+40 Mesh Pretreated Coal	BR-76-23		BR-76-24				
Lime/Coal Feed Ratio			0:1		2:1				
Coal	W. Ky. No. 9		Pretreated W. Ky. No. 9						
Heating Rate, °F/min			5		5				
Terminal Temperature, °F		750	1300		1300				
Holding Time, min		30	60		60				
			Feed	Residue	Feed	+50	−50	Float	Sink
Sulfur, wt %									
Sulfide	0.02	0.04	0.04	0.28	0.01	0.20	0.61	0.09	1.04
Sulfate	0.60	0.10	0.10	0.02	0.03	0.01	0.08	0.04	0.06
Pyritic	1.06	1.54	1.54	0.02	0.52	0.03	0.00	0.01	0.00
Organic	1.82	1.38	1.38	0.74	0.46	0.36	0.22	0.46	0.08
Total	3.50	3.06	3.06	1.06	1.02	0.60	0.91	0.60	1.18
Weight, g									
Initial	100.00		75.00		150				
Treated		90.84		53.3		111.9			
Weight Loss, %									
Total Weight		9.16		28.9		25.4			
Coal Weight		9.16		28.9		76.2			
Reduced Data									
Weight, lb			90.84	64.59	272.52	57.17	146.13	54.89	148.41
Sulfur Weight, lb									
Sulfide	0.02	0.04	0.04	0.18	0.04	0.11	0.89	0.05	1.54
Sulfate	0.60	0.09	0.09	0.01	0.09	0.01	0.12	0.02	0.09
Pyritic	1.06	1.40	1.40	0.48	1.40	0.02	0.00	0.01	0.00
Organic	1.82	1.25	1.25	0.01	1.25	0.21	0.32	0.25	0.12
Total	3.50	2.78	2.78	0.68	2.78	0.35	1.33	0.33	1.75
Sulfur Content %									
Sulfide	0.02	0.04	0.04	--	0.01	--		--	
Sulfate	0.60	0.10	0.10	--	0.03	--		--	
Pyritic	1.06	1.54	1.54	0.02	0.52	0.03		0.01	
Organic	1.82	1.38	1.38	0.74	0.46	0.36		0.46	
Total	3.50	3.06	3.06	0.76	1.02	0.39		0.47	
Sulfur Removal, wt %									
From Feed				82.4		91.7		90.6	
From Coal		20.6		86.0		93.4		92.6	

*Calculated for 40 mesh fraction.

TABLE 26. BATCH REACTOR TEST FOR SO_2 EMISSIONS AND HEATING VALUES, WESTERN KENTUCKY NO. 9 COAL

		lb $SO_2/10^6$ Btu		
	Btu/lb	Total	Pyritic & Organic	Total Heating Value, Btu/100 lb coal feed
Coal (Dry)	12,454	5.62	4.63	1,245,400
Pretreated +40 Mesh	11,809	5.18	4.49	1,072,730
BR-76-1 Residue	12,119	2.06	1.37	805,792
BR-76-2 +50	12,107	0.73	0.53	743,249
Float	12,684	0.20	0.11	735,291
BR-76-3 Residue	11,967	0.85	0.65	749,374
BR-76-4 +50	11,035	0.76	0.34	586,731
Float	12,134	0.71	0.54	602,817
BR-76-5 Residue	11,651	0.96	0.50	582,783
BR-76-6 +50	11,463	0.38	0.16	618,543
Float	12,029	0.48	0.37	577,512
BR-76-12 Residue	11,810	0.99	0.64	750,998
BR-76-22 +50	10,856	0.59	0.41	587,635
Float	12,149	0.49	0.36	674,270
BR-76-23 Residue	11,859	1.79	1.28	765,973
BR-76-24 +50	10,270	1.17	0.76	587,136
Float	11,214	1.07	0.84	615,536

emissions between the lime and no-lime tests. Considering total sulfur, only BR-76-1 and BR-76-23, the lower temperature runs, have emissions that exceed the allowable limit. All the other runs are below the standards even when total sulfur is considered. The higher recovery of material in the no-lime tests will help offset any differences in sulfur removal, because of greater heating value yields.

Figure 15 shows the sulfur content of the treated Western Kentucky No. 9 coal in both the thermobalance and the batch-reactor runs. Although the initial sulfur contents of the feed coals differ slightly, both test series show good sulfur removal.

Rapid Heat-Up Runs

A series of runs with pretreated Western Kentucky No. 9 coal was made in the batch reactor using heat-up rates over 60°F/min (Table 27). This was accomplished by heating the furnace to near 1500°F and then placing the reactor into it. Residence time at the final temperatures was varied from 30 to 330 minutes.

The first two tests, BR-76-7 and BR-76-8, (one with lime acceptor and one without) were held for 5-1/2 hours at the terminal temperature. This represents total run times of equal length for the rapid and slow heat-up tests. These tests resulted in low sulfur values in the treated coal fraction. There is also more material loss, as would be expected from the length of time at 1500°F. The remainder of the runs were made to establish the residence time necessary at the various final temperatures. Examination of the data shows increased sulfur removal as residence time and temperature are increased. The lime tests show a little better removal, on the average, relative to the no-lime tests, but this is offset by much more material recovery in the no-lime test.

Figure 16 represents the test results graphically. The pyritic and organic sulfur content (expressed as a percentage of the original coal sulfur) are plotted against the effective run time (time in excess of 750°F). The effects of terminal temperature and residence time are evident. Each temperature approaches a maximum sulfur removal as the run time increases.

Table 28 lists the calculated sulfur emissions for the direct combustion of the desulfurized material from each of the batch tests. The lower heat-up tests show acceptable levels, considering both total sulfur and the sum of pyritic and organic sulfur, for all tests except BR-76-23. This test was made at only 1300°F and, although the test conditions were the same as BR-76-24, the treated coal was not subjected to the separation techniques that would remove some of the sulfur (because lime was not used and no lime-coal separation was required).

Rapid heat-up tests show acceptable emissions for pyritic and organic sulfur for tests above 1300°F and/or over 60 minutes holding time. Reduction of total sulfur below the emission limitation requires treatment at 1500°F and 60 minutes or longer.

Unpretreated Western Kentucky No. 9

Table 29 lists data, evaluating the effects of pretreatment, from batch reactor tests using Western Kentucky No. 9 coal. Tests BR-76-19 and BR-76-20 were made with unpretreated coal feed. The other two runs, BR-76-29 and BR-76-27, are included for comparison because run conditions are the same. Operation with the unpretreated coal was poor; caking resulted and some material stuck to the reactor walls. The sulfur

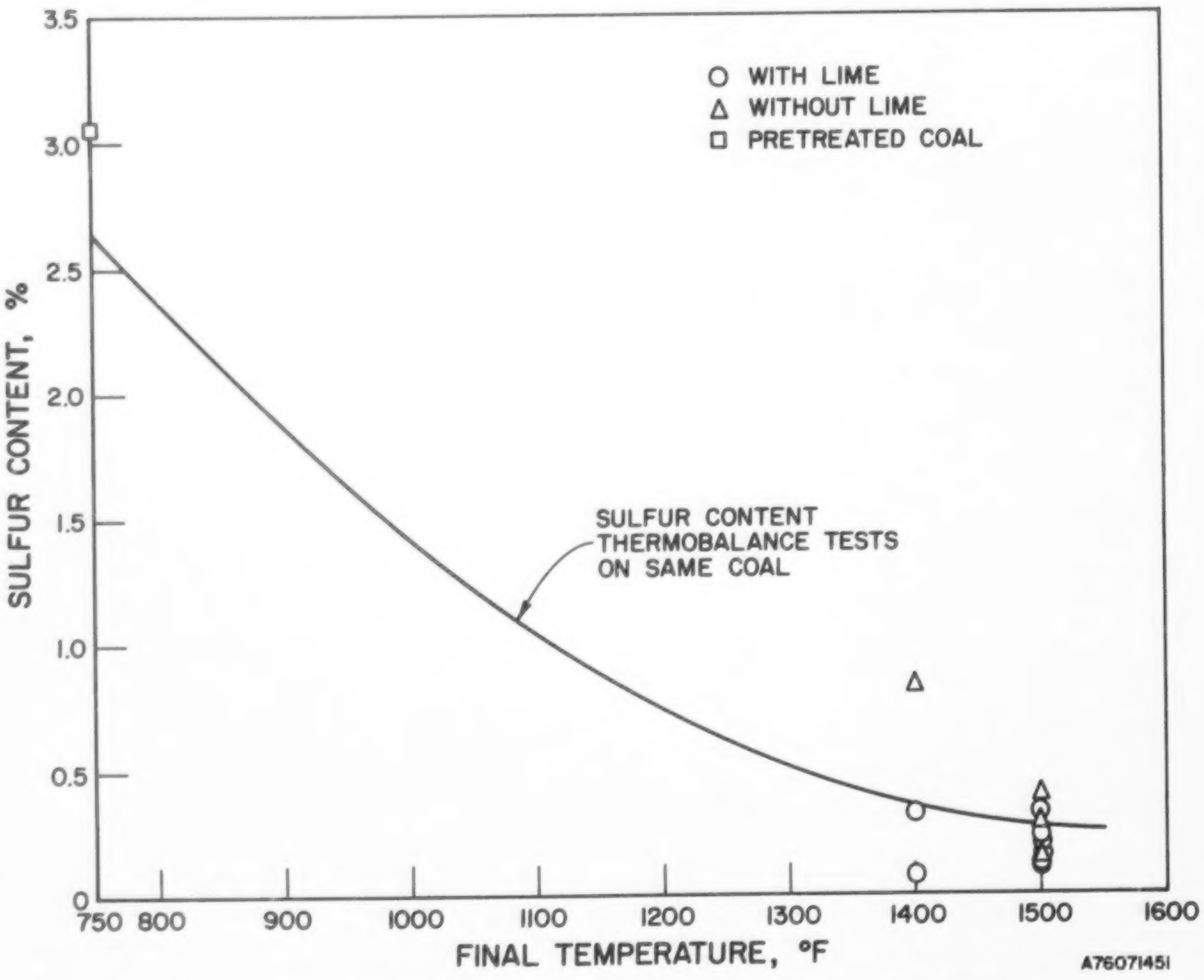

Figure 15. Treated material sulfur content — batch reactor tests on Western Kentucky No. 9 coal.

TABLE 27. BATCH REACTOR RUN DATA FOR PRETREATED WESTERN KENTUCKY NO. 9 COAL

Run No.	Feed Coal*	+40 Mesh Pretreated Coal	BR-76-7		BR-76-8				
			Feed	Residue	Feed	+50	−50	Float	Sink
Lime/Coal Feed Ratio			0:1		2:1				
Coal	W. Ky. No. 9		Pretreated W. Ky. No. 9						
Heating Rate, °F/min			Rapid		Rapid				
Terminal Temperature, °F		750	1500		1500				
Holding Time, min		30	330		330				
Sulfur, wt %									
Sulfide	0.02	0.04	0.04	0.22	0.01	0.13	1.26	0.12	1.35
Sulfate	0.60	0.10	0.10	0.00	0.03	0.01	0.01	0.02	0.03
Pyritic	1.06	1.54	1.54	0.02	0.52	0.00	0.00	0.02	0.00
Organic	1.82	1.38	1.38	0.14	0.46	0.12	0.03	0.14	0.03
Total	3.50	3.06	3.06	0.38	1.02	0.26	1.30	0.30	1.41
Weight, g									
Initial	100.00		75		150				
Treated		90.84		51.2		108.4			
Weight Loss, %									
Total Weight		9.16		31.7		27.7			
Coal Weight		9.16		31.7		83.1			
Reduced Data									
Weight, lb			90.84	62.01	272.52	40.81	156.13	35.71	161.23
Sulfur Weight, lb									
Sulfide	0.02	0.04	0.04	0.14	0.04	0.05	1.96	0.04	2.18
Sulfate	0.60	0.09	0.09	0.00	0.09	0.00	0.02	0.01	0.05
Pyritic	1.06	1.40	1.40	0.01	1.40	0.00	0.00	0.01	0.00
Organic	1.82	1.25	1.25	0.09	1.25	0.05	0.05	0.05	0.05
Total	3.50	2.78	2.78	0.24	2.78	0.10	2.03	0.11	2.28
Sulfur Content %									
Sulfide	0.02	0.04	0.04		0.01	--			
Sulfate	0.60	0.10	0.10		0.03	--			
Pyritic	1.06	1.54	1.54	0.02	0.52	0.00		0.02	
Organic	1.82	1.38	1.38	0.14	0.46	0.12		0.14	
Total	3.50	3.06	3.06	0.16	1.02	0.12		0.16	
Sulfur Removal, wt %									
From Feed				96.4		98.2		97.8	
From Coal		20.6		97.1		98.6		98.3	

*Calculated for 40 mesh fraction.

TABLE 27. BATCH REACTOR RUN DATA FOR PRETREATED WESTERN KENTUCKY NO. 9 COAL (Continued)

Run No.	Feed Coal*	+40 Mesh Pretreated Coal	BR-76-25		BR-76-26		BR-76-27	
Lime/Coal Feed Ratio			0:1		0:1		0:1	
Coal	W. Ky. No. 9		Pretreated W. Ky. No. 9					
Heating Rate, °F/min			Rapid		Rapid		Rapid	
Terminal Temperature, °F		750	1400		1400		1500	
Holding Time, min		30	60		120		60	
			Feed	Residue	Feed	Residue	Feed	Residue
Sulfur, wt %								
Sulfide	0.02	0.04	0.04	0.38	0.04	0.24	0.04	0.47
Sulfate	0.60	0.10	0.10	0.01	0.10	0.01	0.10	0.00
Pyritic	1.06	1.54	1.54	0.03	1.54	0.04	1.54	0.02
Organic	1.82	1.38	1.38	0.63	1.38	0.42	1.38	0.43
Total	3.50	3.06	3.06	1.05	3.06	0.71	3.06	0.92
Weight, g								
Initial	100.00		75.0		75.0		75.0	
Treated		90.84		53.9		53.2		53.4
Weight Loss, %								
Total Weight		9.16		28.13		29.1		28.8
Coal Weight		9.16		28.13		29.1		28.8
Reduced Data								
Weight, lb			90.84	65.29	90.84	64.41	90.84	64.68
Sulfur Weight, lb								
Sulfide	0.02	0.04	0.04	0.25	0.04	0.15	0.04	0.30
Sulfate	0.60	0.09	0.09	0.01	0.09	0.01	0.09	0.00
Pyritic	1.06	1.40	1.40	0.02	1.40	0.03	1.40	0.01
Organic	1.82	1.25	1.25	0.41	1.25	0.27	1.25	0.28
Total	3.50	2.78	2.78	0.69	2.78	0.46	2.78	0.59
Sulfur Content %								
Sulfide	0.02	0.04	0.04	--	0.04	--	0.04	--
Sulfate	0.60	0.10	0.10	--	0.10	--	0.10	--
Pyritic	1.06	1.54	1.54	0.03	1.54	0.04	1.54	0.02
Organic	1.82	1.38	1.38	0.63	1.38	0.42	1.38	0.43
Total	3.50	3.06	3.06	0.68	3.06	0.46	3.06	0.45
Sulfur Removal, wt %								
From Feed				84.5		89.2		89.6
From Coal		20.6		87.7		91.4		91.7

*Calculated for 40 mesh fraction.

TABLE 27. BATCH REACTOR RUN DATA FOR PRETREATED WESTERN KENTUCKY NO. 9 COAL (Continued)

Run No.	Feed Coal*	+40 Mesh Pretreated Coal	BR-76-28					BR-76-29	
Lime/Coal Feed Ratio			2:1					0:1	
Coal	W. Ky. No. 9		Pretreated W. Ky. No. 9						
Heating Rate, °F/min			Rapid					Rapid	
Terminal Temperature, °F		750	1500					1500	
Holding Time, min		30	60					30	
			Feed	+50	−50	Float	Sink	Feed	Residue
Sulfur, wt %									
Sulfide	0.02	0.04	0.01	0.13	1.25	0.13	1.55	0.04	0.47
Sulfate	0.60	0.10	0.03	0.03	0.10	0.04	0.14	0.10	0.02
Pyritic	1.06	1.54	0.52	0.02	0.00	0.02	0.00	1.54	0.01
Organic	1.82	1.38	0.46	0.46	0.39	0.32	0.11	1.38	0.64
Total	3.50	3.06	1.02	0.64	1.74	0.51	1.80	3.06	1.14
Weight, g									
Initial	100.00		150					75	
Treated		90.84		106.3					54.2
Weight Loss, %									
Total Weight		9.16		29.13					27.73
Coal Weight		9.16		87.39					27.73
Reduced Data									
Weight, lb			272.52	57.28	135.85	57.55	135.58	90.84	65.65
Sulfur Weight, lb									
Sulfide	0.02	0.04	0.04	0.07	1.70	0.07	2.10	0.04	0.31
Sulfate	0.60	0.09	0.09	0.02	0.14	0.02	0.19	0.09	0.01
Pyritic	1.06	1.40	1.40	0.01	0.00	0.01	0.00	1.40	0.01
Organic	1.82	1.25	1.25	0.26	0.53	0.18	0.15	1.25	0.42
Total	3.50	2.78	2.78	0.36	2.37	0.28	2.44	2.78	0.75
Sulfur Content %									
Sulfide	0.02	0.04	0.01	--		--		0.04	--
Sulfate	0.60	0.10	0.03	--		--		0.10	--
Pyritic	1.06	1.54	0.52	0.02		0.02		1.54	0.01
Organic	1.82	1.38	0.46	0.46		0.32		1.38	0.64
Total	3.50	3.06	1.02	0.48		0.34		3.06	0.65
Sulfur Removal, wt %									
From Feed				90.3		93.2			84.5
From Coal		20.6		92.3		94.6			87.7

*Calculated for 40 mesh fraction.

TABLE 27. BATCH REACTOR RUN DATA FOR PRETREATED WESTERN KENTUCKY NO. 9 COAL (Continued)

Run No.	Feed Coal*	+40 Mesh Pretreated Coal	BR-76-30		BR-76-31		BR-76-32	
Lime/Coal Feed Ratio			0:1		0:1		0:1	
Coal	W. Ky. No. 9		Pretreated W. Ky. No. 9					
Heating Rate, °F/min			Rapid		Rapid		Rapid	
Terminal Temperature, °F		750	1500		1300		1300	
Holding Time, min		30	120		60		120	
			Feed	Residue	Feed	Residue	Feed	Residue
Sulfur, wt %								
Sulfide	0.02	0.04	0.04	0.22	0.04	0.66	0.04	0.46
Sulfate	0.60	0.10	0.10	0.00	0.10	0.01	0.10	0.01
Pyritic	1.06	1.54	1.54	0.03	1.54	0.03	1.54	0.02
Organic	1.82	1.38	1.38	0.29	1.38	0.81	1.38	0.50
Total	3.50	3.06	3.06	0.54	3.06	1.51	3.06	0.99
Weight, g								
Initial	100.00		75		75		75	
Treated		90.84		52.4		55.2		54.9
Weight Loss, %								
Total Weight		9.16		30.13		26.4		26.8
Coal Weight		9.16		30.13		26.4		26.8
Reduced Data								
Weight, lb			90.84	63.47	90.84	66.86	90.84	66.49
Sulfur Weight, lb								
Sulfide	0.02	0.04	0.04	0.14	0.04	0.44	0.04	0.31
Sulfate	0.60	0.09	0.09	0.00	0.09	0.01	0.09	0.01
Pyritic	1.06	1.40	1.40	0.02	1.40	0.02	1.40	0.01
Organic	1.82	1.25	1.25	0.18	1.25	0.54	1.25	0.33
Total	3.50	2.78	2.78	0.34	2.78	1.01	2.78	0.66
Sulfur Content %								
Sulfide	0.02	0.04	0.04	--	0.04	--	0.04	--
Sulfate	0.60	0.10	0.10	--	0.10	--	0.10	--
Pyritic	1.06	1.54	1.54	0.03	1.54	0.03	1.54	0.02
Organic	1.82	1.38	1.38	0.29	1.38	0.81	1.38	0.50
Total	3.50	3.06	3.06	0.32	3.06	0.84	3.06	0.52
Sulfur Removal, wt %								
From Feed				92.8		79.9		87.8
From Coal		20.6		94.3		84.0		90.3

*Calculated for 40 mesh fraction.

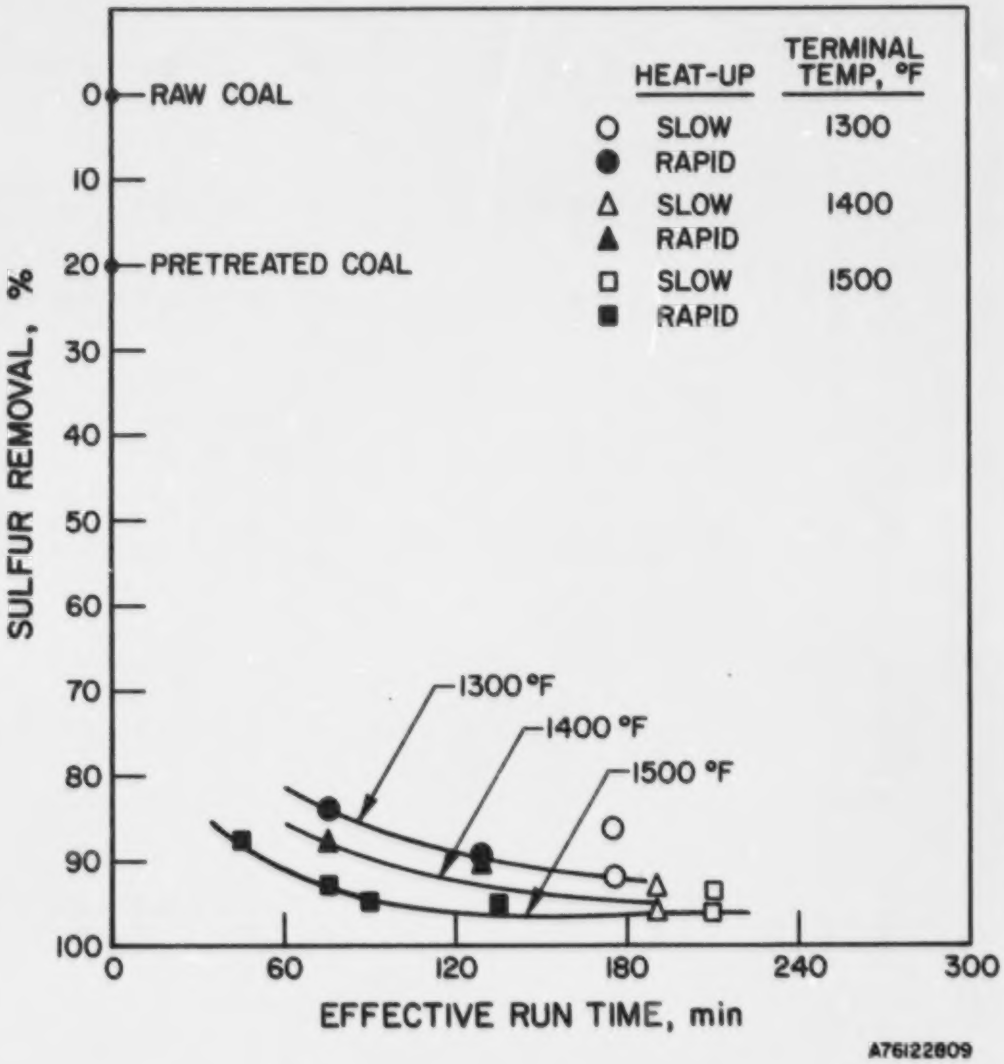

Figure 16. Degree of sulfur removal in batch reactor tests as a function of effective run time.

TABLE 28. SO_2 EMISSIONS AND HEATING VALUES FOR WESTERN KENTUCKY NO. 9 COAL TREATED MATERIAL

	Btu/lb	lb $SO_2/10^6$ Btu Total	 Pyritic & Organic	Total Heating Value, Btu/100 lb Coal Feed
Coal (Dry)	12,454	5.62	4.63	1,245,400
Pretreated +40 Mesh	11,809	5.18	4.95	1,072,730
BR-76-7 Residue	11,565	0.66	0.28	717,146
BR-76-8 +50	9,233	0.56	0.26	376,799
Float	11,789	0.51	0.27	420,985
BR-76-25 Residue	10,898	1.93	1.25	711,530
BR-76-26 Residue	10,908	1.30	0.84	702,584
BR-76-27 Residue	10,693	1.72	0.84	691,623
BR-76-28 +50	10,741	1.19	0.90	615,244
Float	10,679	0.96	0.64	614,576
BR-76-29 Float	10,716	2.13	1.21	703,505
BR-76-30 Float	11,086	0.97	0.58	703,628
BR-76-31 Float	11,159	2.71	1.51	746,091
BR-76-32 Float	11,080	1.79	0.94	736,709

TABLE 29. COMPARISON OF BATCH REACTOR RUNS WITH PRETREATED AND UNPRETREATED WESTERN KENTUCKY NO. 9 COAL

	Feed Coal*	Pretreated Coal*	BR-76-19†		BR-76-29	
Lime/Coal Feed Ratio			0:1		0:1	
Heating Rate, °F/min			Rapid		Rapid	
Terminal Temperature, °F		750	1500		1500	
Holding Time, min		30	30		30	
Sulfur, wt %			Feed	Residue	Feed	Residue
Sulfide	0.02	0.04	0.02	0.27	0.04	0.47
Sulfate	0.60	0.10	0.60	0.01	0.10	0.02
Pyritic	1.06	1.54	1.06	0.05	1.54	0.01
Organic	1.82	1.38	1.82	0.92	1.38	0.64
Total	3.50	3.06	3.50	1.25	3.06	1.14
Weight, g						
Initial	100.00		75.0		75.0	
Treated		90.84		32.2		54.2
Weight Loss, %						
Total Weight		9.16		57.07		27.73
Coal Weight		9.16		57.07		27.73
Reduced Data						
Weight, lb	100.00	90.84	100.00	43.93	90.84	65.65
Sulfur Weight, lb						
Sulfide	0.02	0.04	0.02	0.12	0.04	0.31
Sulfate	0.60	0.09	0.60	0.00	0.09	0.01
Pyritic	1.06	1.40	1.06	0.02	1.40	0.01
Organic	1.82	1.25	1.82	0.40	1.25	0.42
Total	3.50	2.78	3.50	0.54	2.78	0.75
Sulfur Content, %						
Sulfide	0.02	0.04	0.02	-	0.04	-
Sulfate	0.60	0.10	0.60	-	0.10	-
Pyritic	1.06	1.54	1.06	0.05	1.54	0.01
Organic	1.82	1.38	1.82	0.92	1.38	0.64
Total	3.50	3.06	3.50	0.97	3.06	0.65
Sulfur Removal, wt %						
From Feed		20.6		84.57		84.5
From Coal						87.7

* Calculated for +40 mesh fraction.

† Coal feed not pretreated.

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TABLE 29. COMPARISON OF BATCH REACTOR RUNS WITH PRETREATED AND UNPRETREATED WESTERN KENTUCKY NO. 9 COAL (Continued)

	Feed Coal*	Pretreated Coal*	BR-76-20†		BR-76-27	
Lime/Coal Feed Ratio			0:1		0:1	
Heating Rate, °F/min			Rapid		Rapid	
Terminal Temperature, °F		750	1500		1500	
Holding Time, min		30	60		60	
			Feed	Residue	Feed	Residue
Sulfur, wt %						
Sulfide	0.02	0.04	0.02	0.36	0.04	0.47
Sulfate	0.60	0.10	0.60	0.02	0.10	0.00
Pyritic	1.06	1.54	1.06	0.06	1.54	0.02
Organic	1.82	1.38	1.82	0.59	1.38	0.43
Total	3.50	3.06	3.50	1.03	3.06	0.92
Weight, g						
Initial	100.00		75.0		75.0	
Treated		90.84		43.5		53.4
Weight Loss, %						
Total Weight		9.16		42.0		28.8
Coal Weight		9.16		42.0		28.8
Reduced Data						
Weight, lb	100.00	90.84	100.00	58.0	90.84	64.68
Sulfur Weight, lb						
Sulfide	0.02	0.04	0.02	0.21	0.04	0.30
Sulfate	0.60	0.09	0.60	0.01	0.09	0.00
Pyritic	1.06	1.40	1.06	0.03	1.40	0.01
Organic	1.82	1.25	1.82	0.34	1.25	0.28
Total	3.50	2.78	3.50	0.59	2.78	0.59
Sulfur Content, %						
Sulfide	0.02	0.04	0.02	-	0.04	-
Sulfate	0.60	0.10	0.60	-	0.10	-
Pyritic	1.06	1.54	1.06	0.06	1.54	0.02
Organic	1.82	1.38	1.82	0.59	1.38	0.43
Total	3.50	3.06	3.50	0.65	3.06	0.45
Sulfur Removal, wt %						
From Feed		20.6		89.43		89.6
From Coal						91.7

* Calculated for +40 mesh fraction.

† Coal feed not pretreated.

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contents in Table 29 show that the residual organic sulfur in the unpretreated coal tests is much higher than the others. Treated material recovery is less in the unpretreated runs. Results of both sulfur removal and material recovery substantiate the need for adequate coal pretreatment.

Siderite Acceptor Runs

Analyses of batch reactor runs BR-76-9 to BR-76-11 are shown in Table 30. Sintered siderite (FeO), rather than lime, was used as an acceptor in these runs. Conditions were similar to the previous pretreated Western Kentucky No. 9 coal runs, with lime, for comparisons. Data for the comparable lime-acceptor tests are shown in Table 31. The results with the iron acceptor are similar to the results of the lime runs in both reduced sulfur values and materials recovery.

There was difficulty in both operation and laboratory analysis with these runs. The heavier siderite was not amenable to good fluidization and mixing. The additional iron is a detriment in our sulfur-by-types analysis because the pyritic sulfur analysis is made on the iron determination. Great care must be taken so that errors are not made. The siderite appears to be as satisfactory as the lime for an acceptor material. It has the disadvantage of poor mixing and difficulty of analysis. One significant advantage is that it can be regenerated with steam without carbonate formation. The siderite is therefore a feasible candidate for the overall process, should the acceptor concept be desirable.

Nitrogen-Hydrogen Mixtures

Four batch reactor runs were made with mixtures of nitrogen and hydrogen to determine baseline conditions and if nitrogen could be used for the fluidizing control gas, as was done in pretreatment runs.

The results for these runs are shown in Table 32 and graphically represented in Figure 17. The tests used concentrations of hydrogen at 5%, 10%, 20%, and 50% with the balance nitrogen. One previous run with 100% hydrogen is included in the table and graph.

Results show that the hydrogen concentration must be at least 50% to reduce sulfur content adequately. The organic sulfur reduction is the controlling factor because the pyritic sulfur has been nearly eliminated in all the runs, regardless of the hydrogen concentration.

Hydrogen-Carbon Monoxide-Water Mixtures

Batch runs were attempted using mixtures of H_2 and CO. Extremely fine carbon from the reaction –

$$2CO \rightarrow CO_2 + C$$

plugged the porous plate in the reactor. Rapid heat-ups failed to avoid the problem, so water was added to the system.

TABLE 30. BATCH REACTOR RUN DATA, WESTERN KENTUCKY NO. 9 COAL WITH SIDERITE

Run No.	Feed Coal*	Pretreated Coal*	BR-76-9				BR-76-10				
			Feed	+50	−50	Total	Feed	+50	−50	Float	Sink
Siderite/Coal Feed Ratio			2:1				2:1				
Heating Rate, °F/min			15				5				
Terminal Temperature, °F		750	1500				1500				
Holding Time, Min		30	230				30				
Sulfur, wt %											
Sulfide	0.02	0.04	0.01	0.12	1.06	0.79	0.01	0.10	0.74	0.03	0.74
Sulfate	0.60	0.10	0.03	0.00	0.01	0.00	0.03	0.00	0.05	0.00	0.03
Pyritic	1.06	1.54	0.52	0.00	0.06	0.01	0.52	0.01	0.09	0.01	0.10
Organic	1.82	1.38	0.46	0.13	0.03	0.06	0.46	0.20	0.03	0.20	0.02
Total	3.50	3.06	1.02	0.25	1.16	0.86	1.02	0.31	0.91	0.24	0.89
Weight, g											
Initial	100.00		150				150				
Treated		90.84		114.6				116.2			
Weight Loss, %											
Total Weight		9.16		23.6				22.53			
Coal Weight		9.16		70.8				67.60			
Reduced Data											
Weight, lb			272.52	59.97	148.23	208.20	272.52	61.53	149.59	55.48	155.64
Sulfur Weight, lb											
Sulfide	0.02	0.04	0.04	0.07	1.57	1.64	0.04	0.06	1.11	0.02	1.15
Sulfate	0.60	0.09	0.09	0.00	0.01	0.00	0.09	0.00	0.07	0.00	0.05
Pyritic	1.06	1.40	1.40	0.00	0.09	0.02	1.40	0.01	0.13	0.01	0.16
Organic	1.82	1.25	1.25	0.08	0.04	0.12	1.25	0.12	0.04	0.11	0.03
Total	3.50	2.78	2.78	0.15	1.71	1.78	2.78	0.19	1.35	0.14	1.39
Sulfur Content, %											
Sulfide	0.02	0.04	0.01	0.00			0.01	0.00		0.00	
Sulfate	0.60	0.10	0.03	0.00			0.03	0.00		0.00	
Pyritic	1.06	1.54	0.52	0.00			0.52	0.01		0.01	
Organic	1.82	1.38	0.46	0.13			0.46	0.20		0.20	
Total	3.50	3.06	1.02	0.13			1.02	0.21		0.21	
Sulfur Removal, wt %											
From Feed				97.1				95.3		95.7	
From Coal		20.6		97.7				96.3		96.6	

*Calculated for 40 mesh.

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TABLE 30. BATCH REACTOR RUN DATA, WESTERN KENTUCKY NO. 9 COAL WITH SIDERITE (Continued)

	Feed Coal*	Pretreated Coal*					
Run No.			BR-76-11				
Siderite/Coal Feed Ratio			2:1				
Heating Rate, °F/min			5				
Terminal Temperature, °F		750	1500				
Holding Time, Min		30	30				
Sulfur, wt %			Feed	+50	−50	Float	Sink
Sulfide	0.02	0.04	0.01	0.12	0.53	0.06	0.71
Sulfate	0.60	0.10	0.03	0.01	0.03	0.01	0.03
Pyritic	1.06	1.54	0.52	0.02	0.28	0.01	0.12
Organic	1.82	1.38	0.46	0.22	0.02	0.17	0.03
Total	3.50	3.06	1.02	0.37	0.86	0.25	0.89
Weight, g							
Initial	100.00		150				
Treated		90.84		114.5			
Weight Loss, %							
Total Weight		9.16		23.7			
Coal Weight		9.16		71.0			
Reduced Data							
Weight, lb			272.52	55.17	152.76	53.11	154.82
Sulfur Weight, lb							
Sulfide	0.02	0.04	0.04	0.07	0.81	0.03	1.10
Sulfate	0.60	0.09	0.09	0.01	0.05	0.01	0.05
Pyritic	1.06	1.40	1.40	0.01	0.43	0.01	0.19
Organic	1.82	1.25	1.25	0.12	0.03	0.09	0.05
Total	3.50	2.78	2.78	0.21	1.32	0.14	1.39
Sulfur Content, %							
Sulfide	0.02	0.04	0.01	0.00		0.00	
Sulfate	0.60	0.10	0.03	0.00		0.00	
Pyritic	1.06	1.54	0.52	0.02		0.01	
Organic	1.82	1.38	0.46	0.22		0.17	
Total	3.50	3.06	1.02	0.24		0.18	
Sulfur Removal, wt %							
From Feed				95.3		96.4	
From Coal		20.6		96.3		97.1	

*Calculated for 40 mesh.

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TABLE 31. BATCH REACTOR RUN DATA FOR WESTERN KENTUCKY NO. 9 COAL

Run No.	Feed Coal*	+40 Mesh Pretreated Coal	BR-76-3		BR-76-4				
Lime/Coal Feed Ratio			0:1		2:1				
Coal	W. Ky. No. 9		Pretreated W. Ky. No. 9						
Heating Rate, °F/min			5		5				
Terminal Temperature, °F		750	1500		1500				
Holding Time, min		30	30		30				
			Feed	Residue	Feed	+50	−50	Float	Sink
Sulfur, wt %									
Sulfide	0.02	0.04	0.04	0.12	0.01	0.22	1.29	0.10	1.30
Sulfate	0.60	0.10	0.10	0.00	0.03	0.01	0.05	0.00	0.03
Pyritic	1.06	1.54	1.54	0.02	0.52	0.02	0.00	0.01	0.00
Organic	1.82	1.38	1.38	0.37	0.46	0.17	0.04	0.32	0.08
Total	3.50	3.06	3.06	0.51	1.02	0.42	1.38	0.43	1.41
Weight, g									
Initial	100.00		75.0		150				
Treated		90.84		51.7		121.9			
Weight Loss, %									
Total Weight		9.16		31.1		18.7			
Coal Weight		9.16		31.1		56.2			
Reduced Data									
Weight, lb			90.84	62.62	272.52	53.17	168.29	49.68	171.78
Sulfur Weight, lb									
Sulfide	0.02	0.04	0.04	0.08	0.04	0.12	2.17	0.05	2.23
Sulfate	0.60	0.09	0.09	0.00	0.09	0.01	0.08	0.00	0.05
Pyritic	1.06	1.40	1.40	0.01	1.40	0.01	0.00	0.01	0.00
Organic	1.82	1.25	1.25	0.23	1.25	0.09	0.07	0.16	0.14
Total	3.50	2.78	2.78	0.32	2.78	0.23	2.32	0.22	2.42
Sulfur Content %									
Sulfide	0.02	0.04	0.04		0.01				
Sulfate	0.60	0.10	0.10		0.03				
Pyritic	1.06	1.54	1.54	0.02	0.52	0.02		0.01	
Organic	1.82	1.38	1.38	0.37	0.46	0.17		0.32	
Total	3.50	3.06	3.06	0.39	1.02	0.19		0.33	
Sulfur Removal, wt %									
From Feed				91.4		96.4		93.9	
From Coal		20.6		93.1		97.1		95.1	

*Calculated for 40 mesh fraction.

TABLE 31. BATCH REACTOR RUN DATA FOR WESTERN KENTUCKY NO. 9 COAL (Continued)

Run No.	Feed Coal*	+40 Mesh Pretreated Coal	BR-76-5		BR-76-6				
Lime/Coal Feed Ratio			0:1		2:1				
Coal	W. Ky. No. 9		Pretreated W. Ky. No. 9						
Heating Rate, °F/min			20		20				
Terminal Temperature, °F		750	1500		1500				
Holding Time, min		30	240		240				
			Feed	Residue	Feed	+50	−50	Float	Sink
Sulfur, wt %									
Sulfide	0.02	0.04	0.04	0.26	0.01	0.13	1.02	0.06	0.95
Sulfate	0.60	0.10	0.10	0.01	0.03	0.00	0.01	0.01	0.03
Pyritic	1.06	1.54	1.54	0.02	0.52	0.00	0.00	0.03	0.00
Organic	1.82	1.38	1.38	0.27	0.46	0.09	0.02	0.19	0.02
Total	3.50	3.06	3.06	0.56	1.02	0.22	1.05	0.29	1.00
Weight, g									
Initial	100.00		75		150				
Treated		90.84		41.3		119.4			
Weight Loss, %									
Total Weight		9.16		44.93		20.4			
Coal Weight		9.16		44.93		61.2			
Reduced Data									
Weight, lb			90.84	50.02	272.52	53.96	162.97	48.01	168.92
Sulfur Weight, lb									
Sulfide	0.02	0.04	0.04	0.13	0.04	0.07	1.66	0.03	1.60
Sulfate	0.60	0.09	0.09	0.01	0.09	0.00	0.02	0.00	0.05
Pyritic	1.06	1.40	1.40	0.01	1.40	0.00	0.00	0.01	0.00
Organic	1.82	1.25	1.25	0.14	1.25	0.05	0.03	0.09	0.03
Total	3.50	2.78	2.78	0.29	2.78	0.12	1.71	0.13	1.68
Sulfur Content %									
Sulfide	0.02	0.04	0.04	--	0.01	--		--	
Sulfate	0.60	0.10	0.10	--	0.03	--		--	
Pyritic	1.06	1.54	1.54	0.02	0.52	0.00		0.03	
Organic	1.82	1.38	1.38	0.27	0.46	0.09		0.19	
Total	3.50	3.06	3.06	0.29	1.02	0.09		0.22	
Sulfur Removal, wt %									
From Feed				94.6		98.2		96.4	
From Coal		20.6		95.7		98.6		97.1	

*Calculated for 40 mesh fraction.

TABLE 32. PRETREATED WESTERN KENTUCKY NO. 9 COAL BATCH REACTOR RUNS WITH N_2-H_2 GAS MIXTURES

Run No.	Feed Coal*	Pretreated Coal*	BR-76-3		BR-76-12		BR-76-13	
Lime/Coal Feed Ratio			0:1		0		0	
Heating Rate, °F/min			5		5		5	
Terminal Temperature, °F		750	1500		1500		1500	
Holding Time, min		30	30		30		30	
H_2/N_2 Gas Ratio			100:0		5:95		10:90	
Sulfur, wt %			Feed	Residue	Feed	Residue	Feed	Residue
Sulfide	0.02	0.04	0.04	0.12	0.04	0.80	0.04	0.69
Sulfate	0.60	0.10	0.10	0.00	0.10	0.00	0.10	0.01
Pyritic	1.06	1.54	1.54	0.02	1.54	0.04	1.54	0.03
Organic	1.82	1.38	1.38	0.37	1.38	1.37	1.38	1.17
Total	3.50	3.06	3.06	0.51	3.06	2.21	3.06	1.90
Weight, g								
Initial	100.00		75.0		75.0		75.0	
Treated		90.84		51.7		49.2		54.7
Weight Loss, %								
Total Weight		9.16		31.1		34.4		27.1
Coal Weight		9.16		31.1		34.4		27.1
Reduced Data								
Weight, lb	100.00	90.84	90.84	62.62	90.84	59.59	90.84	66.22
Sulfur Weight, lb								
Sulfide	0.02	0.04	0.04	0.08	0.04	0.48	0.04	0.46
Sulfate	0.60	0.09	0.09	0.00	0.09	0.00	0.09	0.01
Pyritic	1.06	1.40	1.40	0.01	1.40	0.02	1.40	0.02
Organic	1.82	1.25	1.25	0.23	1.25	0.82	1.25	0.77
Total	3.50	2.78	2.78	0.32	2.78	1.32	2.78	1.26
Sulfur Content, %								
Sulfide	0.02	0.04	0.04		0.04		0.04	
Sulfate	0.60	0.10	0.10		0.10		0.10	
Pyritic	1.06	1.54	1.54	0.02	1.54	0.04	1.54	0.03
Organic	1.82	1.38	1.38	0.37	1.38	1.37	1.38	1.17
Total	3.50	3.06	3.06	0.39	3.06	1.41	3.06	1.20
Sulfur Removal, wt %								
From Feed				91.4		69.8		71.6
From Coal		20.6		93.1		76.0		77.4

*Calculated for +40 mesh.

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TABLE 32. PRETREATED WESTERN KENTUCKY NO. 9 COAL BATCH REACTOR RUNS WITH N_2-H_2 GAS MIXTURES (Continued)

Run No.	Feed Coal*	Pretreated Coal*	BR-76-14		BR-76-15	
Lime/Coal Feed Ratio			0		0	
Heating Rate, °F/min			5		5	
Terminal Temperature, °F		750	1500		1500	
Holding Time, min		30	30		30	
H_2/N_2 Gas Ratio			20:80		50:50	
Sulfur, wt %			Feed	Residue	Feed	Residue
Sulfide	0.02	0.04	0.04	0.41	0.04	0.27
Sulfate	0.60	0.10	0.10	0.01	0.10	0.02
Pyritic	1.06	1.54	1.54	0.03	1.54	0.04
Organic	1.82	1.38	1.34	0.99	1.34	0.49
Total	3.50	3.06	3.06	1.44	3.06	0.82
Weight, g						
Initial	100.00		75.0		75.0	
Treated		90.84		55.3		53.2
Weight Loss, %						
Total Weight		9.16		26.3		29.0
Coal Weight		9.16		26.3		29.0
Reduced Data						
Weight, lb	100.00	90.84	90.84	66.95	90.84	64.49
Sulfur Weight, lb						
Sulfide	0.02	0.04	0.04	0.27	0.04	0.17
Sulfate	0.60	0.09	0.09	0.01	0.09	0.01
Pyritic	1.06	1.40	1.40	0.02	1.40	0.03
Organic	1.82	1.25	1.25	0.66	1.25	0.32
Total	3.50	2.78	2.78	0.96	2.78	0.53
Sulfur Content, %						
Sulfide	0.02	0.04	0.04		0.04	
Sulfate	0.60	0.10	0.10		0.10	
Pyritic	1.06	1.54	1.54	0.03	1.54	0.04
Organic	1.82	1.38	1.38	0.99	1.38	0.49
Total	3.50	3.06	3.06	1.02	3.06	0.53
Sulfur Removal, wt %						
From Feed				75.5		87.4
From Coal		20.6		80.6		90.0

*Calculated for +40 mesh.

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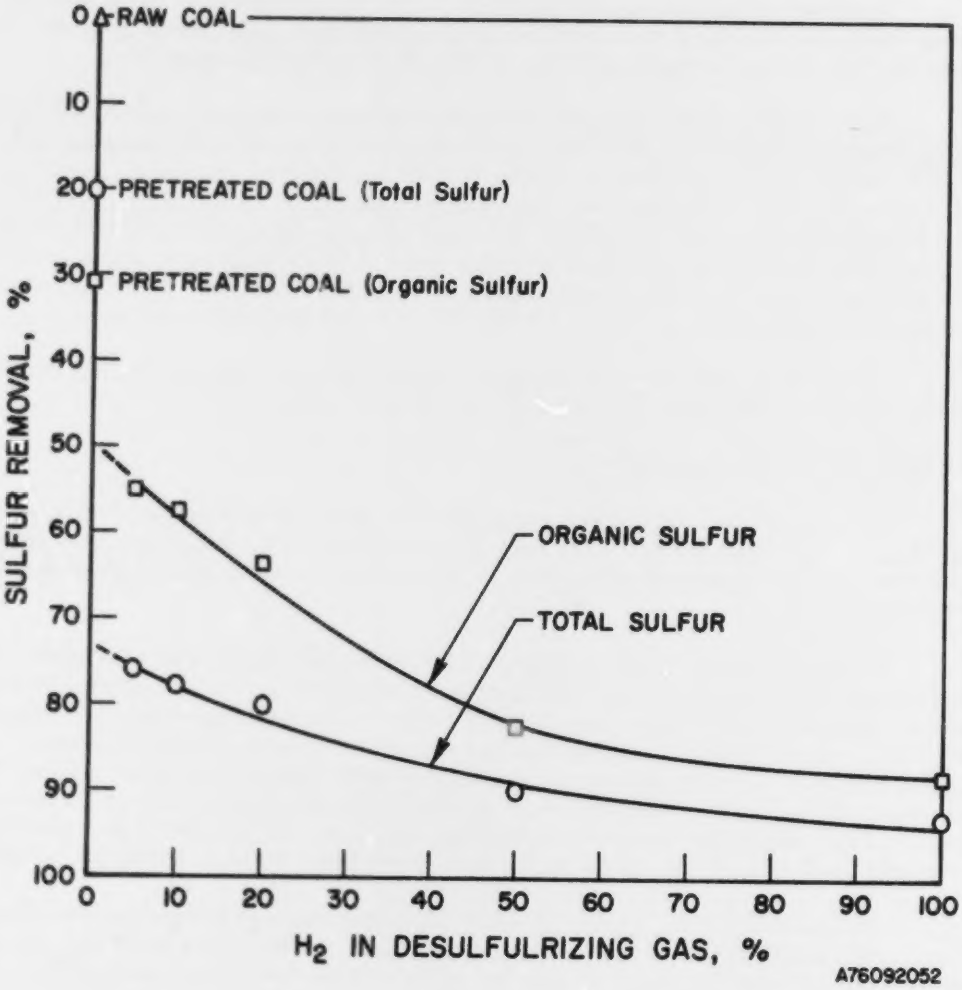

Figure 17. Effect of hydrogen on sulfur removal.

Two runs, BR-76-45 and BR-76-46, were accomplished using the H_2-CO-H_2O mixture. Concentrations of CO over 37.5% precipitate enough carbon to plug the diffusion plate. Data from the two runs are shown in Table 33. The test with a low CO concentration yielded results similar to a previous batch reactor run with hydrogen at the same conditions. The second run gave higher sulfur values, but in both runs the treated material yields an acceptable SO_2 emission when burned.

1% H_2S in Hydrogen

Analysis of the conceptual flow sheet for the process indicated that the off-gas from the hydrodesulfurization could contain significant H_2S concentrations. Consequently, batch reactor tests were made with H_2S in the hydrogen feed.

Table 34 shows the comparison of results from tests using pure hydrogen and those using 9800 ppm (0.98%) H_2S in hydrogen. The two tests with H_2S-bearing gas, H_2S-1 and H_2S-2, were made at conditions similar to two previous batch reactor runs that did not contain H_2S in the feed gas. The first pair of runs in the table, H_2S-1 and BR-76-27, were both heated rapidly to 1500°F and then held for 60 minutes. Sulfide-type sulfur is much higher for H_2S-1, and organic sulfur has not been reduced as much as in BR-76-27. The second pair of runs were heated rapidly to 1400°F and held for 60 minutes. Sulfur values are higher for H_2S-2 than for BR-76-25 at the same conditions.

These tests indicate that a high back pressure of hydrogen sulfide inhibits the desulfurization of the coal. The effect is greater at lower temperature.

ILLINOIS NO. 6 COAL RUNS

Results and run conditions for a series of batch reactor tests made with −10+40 mesh pretreated Illinois No. 6 coal are shown in Table 35. Sulfur removal is increased as the final temperature or holding time is increased. Material recovery from these tests is excellent.

A second series of tests, shown in Table 36, was made with the same conditions, but the pretreated Illinois No. 6 coal was not screened before the tests. This was done to see if the coal particle size was a factor in the sulfur removal step. The sample for BR-76-38 was accidentally discarded before sulfur-by-type was determined, but total sulfur shows it to follow the trends associated with temperature and holding time. Comparison between these tests and those of Table 35 show essentially no difference in sulfur removal and slight differences in material recovery. These results indicate that sulfur removal from the −40 mesh material is no more difficult than from the +40 mesh.

The last four runs, shown in Table 37, were made with screened, pretreated Illinois No. 6 coal mixed with lime. Run conditions again are the same as in the two previous sets. There is slightly better sulfur removal in these tests than the previous two, probably due to the separation step. The lower sulfur is offset by less material recovery and some carbon loss to the −50 mesh (lime) material.

Calculated SO_2 emissions and heating values for the treated material from the 12 runs are shown in Table 38. The emissions for all are below the 1.2 lb SO_2/million Btu limit for total sulfur. The total heating value is higher for the runs without lime.

TABLE 33. BATCH REACTOR RUN DATA

			Run No.					
	Feed Coal	Pretreated Coal, +40 mesh	BR-76-7		BR-76-45*		BR-76-46**	
Coal Type	W. Ky. No. 9	W. Ky. No. 9	Pretreated W. Ky. No. 9		Pretreated W. Ky. No. 9		Pretreated W. Ky. No. 9	
Heating Rate, °F/min	--	--	Rapid		Rapid		Rapid	
Terminal Temperature, °F	--	750	1500		1500		1500	
Holding Time, min	--	30	330		330		330	
			Feed	Residue	Feed	Residue	Feed	Residue
Sulfur, wt %								
Sulfide	0.02	0.04	0.04	0.22	0.04	0.04	0.04	0.09
Sulfate	0.60	0.10	0.10	0.00	0.10	0.00	0.10	0.00
Pyritic	1.06	1.54	1.54	0.02	1.54	0.05	1.54	0.04
Organic	1.82	1.38	1.38	0.14	1.38	0.07	1.38	0.19
Total	3.50	3.06	3.06	0.38	3.06	0.16	3.06	0.32
Weight, g								
Initial	100.00	--	75	--	50	--	50	--
Treated	--	90.84	--	51.2	--	32	--	36.6
Weight Loss, %								
Total Weight	--	9.16	--	31.7	--	36.0	--	26.8
Coal Weight	--	9.16	--	31.7	--	36.0	--	26.8
Reduced Data								
Weight, lb	--	--	90.84	62.01	90.84	58.1	90.84	66.5
Sulfur Weight, lb								
Sulfide	0.02	0.04	0.04	0.14	0.04	0.02	0.04	0.06
Sulfate	0.60	0.09	0.09	0.00	0.09	0.00	0.09	0.00
Pyritic	1.06	1.40	1.40	0.01	1.40	0.03	1.40	0.03
Organic	1.82	1.25	1.25	0.09	1.25	0.04	1.25	0.13
Total	3.50	2.78	2.78	0.24	2.78	0.09	2.78	0.22
Sulfur Content, %								
Sulfide	0.02	0.04	0.04	--	0.04	--	0.04	--
Sulfate	0.60	0.10	0.10	--	0.10	--	0.10	--
Pyritic	1.06	1.54	1.54	0.02	1.54	0.05	1.54	0.04
Organic	1.82	1.38	1.38	0.14	1.38	0.07	1.25	0.19
Total	3.50	3.06	3.06	0.16	3.06	0.12	2.93	0.23
Sulfur Removal, wt %								
From Feed	--	--	--	96.4	--	97.5	--	94.2
From Coal	--	20.6	--	97.1	--	98.0	--	95.4

* Gas composition: 25% H_2O, 62.5% H_2, 12.5% CO.

** Gas composition: 25% H_2O, 37.5% H_2, 37.5% CO.

B77051092

TABLE 34. COMPARISON OF SULFUR VALUES FOR H_2 AND H_2-H_2S RUNS

Run No.	H_2S-1	BR-76-27	H_2S-2	BR-76-25
Heating Rate, °F/min	Rapid	Rapid	Rapid	Rapid
Terminal Temperature, °F	1500	1500	1400	1400
Holding Time, min	60	60	60	60
Sulfur, wt %				
Sulfide	0.83	0.47	0.65	0.38
Sulfate	0.00	0.00	0.03	0.01
Pyritic	0.03	0.02	0.06	0.03
Organic	0.75	0.43	1.48	0.63
Total	1.61	0.92	2.22	1.05

TABLE 35. BATCH REACTOR TESTS – PRETREATED ILLINOIS NO. 6 COAL (–10 +40 MESH)

	Feed Coal*	Pretreated Coal*	BR-76-33		BR-76-34	
			Feed	Residue	Feed	Residue
Lime/Coal Feed Ratio			0:1		0:1	
Heating Rate, °F/min			5		5	
Terminal Temperature, °F		750	1400		1500	
Holding Time, min		30	30		30	
Sulfur, wt %						
Sulfide	0.01	0.01	0.01	0.04	0.01	0.05
Sulfate	0.13	0.04	0.04	0.01	0.04	0.05
Pyritic	0.84	0.65	0.65	0.03	0.65	0.03
Organic	1.50	1.52	1.52	0.56	1.52	0.47
Total	2.48	2.22	2.22	0.64	2.22	0.60
Weight, g						
Initial	100.00		75.0		75.0	
Treated		90.4		54.2		52.2
Weight Loss, %						
Total Weight		9.6		27.7		30.4
Coal Weight		9.6		27.7		30.4
Reduced Data						
Weight, lb	100.00	90.4	90.4	65.4	90.4	62.9
Sulfur Weight, lb						
Sulfide	0.01	0.01	0.01	0.03	0.01	0.03
Sulfate	0.13	0.04	0.04	0.01	0.04	0.03
Pyritic	0.84	0.59	0.59	0.02	0.59	0.02
Organic	1.50	1.37	1.37	0.37	1.37	0.30
Total	2.48	2.01	2.01	0.43	2.01	0.38
Sulfur Content, %						
Sulfide	0.01	0.01	0.01		0.01	
Sulfate	0.13	0.04	0.04		0.04	
Pyritic	0.84	0.65	0.65	0.03	0.65	0.03
Organic	1.50	1.52	1.52	0.56	1.52	0.47
Total	2.48	2.22	2.22	0.59	2.22	0.50
Sulfur Removal, wt %						
From Feed				80.6		84.1
From Coal		18.9		84.3		87.1

*Calculated for +40 mesh fraction.

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TABLE 35. BATCH REACTOR TESTS – PRETREATED ILLINOIS NO. 6 COAL (–10 +40 MESH)
(Continued)

	Feed Coal*	Pretreated Coal*	BR-76-35		BR-76-36	
Lime/Coal Feed Ratio			0:1		0:1	
Heating Rate, °F/min			20		Rapid	
Terminal Temperature, °F		750	1500		1500	
Holding Time, min		30	120		240	
Sulfur, wt %			Feed	Residue	Feed	Residue
Sulfide	0.01	0.01	0.01	0.03	0.01	0.03
Sulfate	0.13	0.04	0.04	0.00	0.04	0.00
Pyritic	0.84	0.65	0.65	0.03	0.65	0.04
Organic	1.50	1.52	1.52	0.44	1.52	0.35
Total	2.48	2.22	2.22	0.50	2.22	0.42
Weight, g						
Initial	100.00		75.0		75.0	
Treated		90.4		61.8		53.6
Weight Loss, %						
Total Weight		9.6		17.6		28.5
Coal Weight		9.6		17.6		28.5
Reduced Data						
Weight, lb	100.00	90.4	90.4	74.5	90.4	64.6
Sulfur Weight, lb						
Sulfide	0.01	0.01	0.01	0.02	0.01	0.02
Sulfate	0.13	0.04	0.04	0.00	0.04	0.00
Pyritic	0.84	0.59	0.59	0.02	0.59	0.03
Organic	1.50	1.37	1.37	0.33	1.37	0.23
Total	2.48	2.01	2.01	0.37	2.01	0.28
Sulfur Content, %						
Sulfide	0.01	0.01	0.01		0.01	
Sulfate	0.13	0.04	0.04		0.04	
Pyritic	0.84	0.65	0.65	0.03	0.65	0.04
Organic	1.50	1.52	1.52	0.44	1.52	0.35
Total	2.48	2.22	2.22	0.47	2.22	0.39
Sulfur Removal, wt %						
From Feed				82.6		87.1
From Coal		18.9		85.9		89.5

*Calculated for +40 mesh fraction.

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TABLE 36. BATCH REACTOR TESTS – PRETREATED ILLINOIS NO. 6 COAL (UNSCREENED)

	Feed Coal	Pretreated Coal	BR-76-37		BR-76-38	
Lime/Coal Feed Ratio			0:1		0:1	
Heating Rate, °F/min			5		5	
Terminal Temperature, °F			1400		1500	
Holding Time, min			30		30	
Sulfur, wt %			Feed	Residue	Feed	Residue
Sulfide	0.01	0.01	0.01	0.06	0.01	
Sulfate	0.13	0.04	0.04	0.00	0.04	
Pyritic	0.83	0.64	0.64	0.04	0.64	
Organic	1.48	1.50	1.50	0.55	1.50	
Total	2.45	2.19	2.19	0.65	2.19	0.64
Weight, g						
Initial	100.00		75.0		75.0	
Treated		90.4		53.4		60.7
Weight Loss, %						
Total Weight				28.8		19.1
Coal Weight				28.8		19.1
Reduced Data						
Weight, lb	100.00	90.4	90.4	64.4	90.4	73.1
Sulfur Weight, lb						
Sulfide	0.01	0.01	0.01	0.04	0.01	
Sulfate	0.13	0.04	0.04	0.00	0.04	
Pyritic	0.83	0.58	0.58	0.03	0.58	
Organic	1.48	1.34	1.34	0.35	1.34	
Total	2.45	1.97	1.97	0.42	1.97	0.47
Sulfur Content, %						
Sulfide	0.01	0.01	0.01		0.01	
Sulfate	0.13	0.04	0.04		0.04	
Pyritic	0.83	0.64	0.64	0.04	0.64	
Organic	1.48	1.50	1.50	0.55	1.50	
Total	2.45	2.19	2.19	0.59	2.19	
Sulfur Removal, wt %						
From Feed				80.7		
From Coal		19.6		81.5		

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TABLE 36. BATCH REACTOR TESTS – PRETREATED ILLINOIS NO. 6 COAL (UNSCREENED) (Continued)

	Feed Coal	Pretreated Coal	BR-76-39		BR-76-40	
Lime/Coal Feed Ratio			0:1		0:1	
Heating Rate, °F/min			20		Rapid	
Terminal Temperature, °F			1500		1500	
Holding Time, min			120		240	
Sulfur, wt %			Feed	Residue	Feed	Residue
Sulfide	0.01	0.01	0.01	0.05	0.01	0.05
Sulfate	0.13	0.04	0.04	0.01	0.04	0.01
Pyritic	0.83	0.64	0.64	0.03	0.64	0.03
Organic	1.48	1.50	1.50	0.41	1.50	0.35
Total	2.45	2.19	2.19	0.50	2.19	0.44
Weight, g						
Initial	100.00		75.0		75.0	
Treated		90.4		53.9		53.0
Weight Loss, %						
Total Weight				28.1		29.3
Coal Weight				28.1		29.3
Reduced Data						
Weight, lb	100.00	90.4	90.4	65.0	90.4	63.9
Sulfur Weight, lb						
Sulfide	0.01	0.01	0.01	0.03	0.01	0.03
Sulfate	0.13	0.04	0.04	0.01	0.04	0.01
Pyritic	0.83	0.58	0.58	0.02	0.58	0.02
Organic	1.48	1.34	1.34	0.27	1.34	0.22
Total	2.45	1.97	1.97	0.33	1.97	0.28
Sulfur Content, %						
Sulfide	0.01	0.01	0.01		0.01	
Sulfate	0.13	0.04	0.04		0.04	
Pyritic	0.83	0.64	0.64	0.03	0.64	0.03
Organic	1.48	1.50	1.50	0.41	1.50	0.35
Total	2.45	2.19	2.19	0.44	2.19	0.38
Sulfur Removal, wt %						
From Feed				85.3		87.8
From Coal		19.6		88.2		90.2

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TABLE 37. BATCH REACTOR TESTS -- PRETREATED ILLINOIS NO. 6 COAL (−10+40 MESH + LIME)

	Feed Coal*	Pretreated Coal*	BR-76-41			BR-76-42		
			Feed	+50	−50	Feed	+50	−50
Lime/Coal Feed Ratio			2:1			2:1		
Heating Rate, °F/min			5			5		
Terminal Temperature, °F		750	1400			1500		
Holding Time, min		30	30			30		
Sulfur, wt %								
Sulfide	0.01	0.01	0.00	0.10	1.27	0.00	0.12	1.10
Sulfate	0.13	0.04	0.01	0.00	0.07	0.01	0.00	0.02
Pyritic	0.84	0.65	0.22	0.03	0.01	0.22	0.02	0.01
Organic	1.50	1.52	0.51	0.43	0.10	0.51	0.27	0.10
Total	2.48	2.22	0.74	0.56	1.45	0.74	0.41	1.23
Weight, g								
Initial	100.00		150.0			150.0		
Treated		90.4		— 111.2 —			— 103.5 —	
Weight Loss, %								
Total Weight		9.6		— 25.9 —			— 31.0 —	
Coal Weight		9.6		— 77.6 —			— 93.0 —	
Reduced Data								
Weight, lb	100.00	90.4	271.20	71.20	129.75	271.20	55.14	131.99
Sulfur Weight, lb								
Sulfide	0.01	0.01	0.01	0.07	1.65	0.01	0.06	1.45
Sulfate	0.13	0.04	0.04	0.00	0.09	0.04	0.00	0.03
Pyritic	0.84	0.59	0.59	0.02	0.01	0.59	0.01	0.01
Organic	1.50	1.37	1.37	0.31	0.13	1.37	0.15	0.13
Total	2.48	2.01	2.01	0.40	1.88	2.01	0.22	1.62
Sulfur Content, %								
Sulfide	0.01	0.01	0.00			0.00		
Sulfate	0.13	0.04	0.01			0.01		
Pyritic	0.84	0.65	0.22	0.03		0.22	0.02	
Organic	1.50	1.52	0.51	0.43		0.51	0.27	
Total	2.48	2.22	0.74	0.46		0.74	0.29	
Sulfur Removal, wt %								
From Feed				83.6			92.0	
From Coal		18.9		86.7			93.5	

*Calculated for +40 mesh fraction.

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TABLE 37. BATCH REACTOR TESTS – PRETREATED ILLINOIS NO. 6 COAL (–10+40 MESH + LIME) (Continued)

	Feed Coal*	Pretreated Coal*	BR-76-43			BR-76-44		
			Feed	+50	–50	Feed	+50	–50
Lime/Coal Feed Ratio			2:1			2:1		
Heating Rate, °F/min			20			Rapid		
Terminal Temperature, °F		750	1500			1500		
Holding Time, min		30	120			240		
Sulfur, wt %								
Sulfide	0.01	0.01	0.00	0.13	1.14	0.00	0.14	1.11
Sulfate	0.13	0.04	0.01	0.01	0.05	0.01	0.01	0.06
Pyritic	0.84	0.65	0.22	0.05	0.02	0.22	0.02	0.01
Organic	1.50	1.52	0.51	0.21	0.12	0.51	0.14	0.25
Total	2.48	2.22	0.74	0.40	1.33	0.74	0.31	1.43
Weight, g								
Initial	100.00		150.0			150.0		
Treated		90.4	— 110.3 —			— 108.1 —		
Weight Loss, %								
Total Weight		9.6	— 26.5 —			— 27.9 —		
Coal Weight		9.6	— 79.4 —			— 83.8 —		
Reduced Data								
Weight, lb	100.00	90.4	271.20	64.51	134.81	271.20	58.42	137.11
Sulfur Weight, lb								
Sulfide	0.01	0.01	0.01	0.08	1.54	0.01	0.08	1.52
Sulfate	0.13	0.04	0.04	0.01	0.07	0.04	0.01	0.08
Pyritic	0.84	0.59	0.59	0.03	0.03	0.59	0.01	0.01
Organic	1.50	1.37	1.37	0.14	0.16	1.37	0.08	0.34
Total	2.48	2.01	2.01	0.26	1.80	2.01	0.18	1.95
Sulfur Content, %								
Sulfide	0.01	0.01	0.00			0.00		
Sulfate	0.13	0.04	0.01			0.01		
Pyritic	0.84	0.65	0.22	0.05		0.22	0.02	
Organic	1.50	1.52	0.51	0.21		0.51	0.14	
Total	2.48	2.22	0.74	0.26		0.74	0.16	
Sulfur Removal, wt %								
From Feed				91.5			95.5	
From Coal		18.9		93.1			96.3	

*Calculated for +40 mesh fraction.

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TABLE 38. SO_2 EMISSIONS AND HEATING VALUES FOR ILLINOIS NO. 6 COAL TREATED MATERIAL

Run No.	Btu/lb	lb $SO_2/10^6$ Btu Total	 Pyritic and Organic	Total Heating Value, Btu/100 lb coal feed
Coal (Dry)	13,022	3.76	3.55	1,302,200
Pretreated (–10+40 mesh)	12,915	3.44	3.36	1,167,516
Pretreated Coal	13,069	3.35	3.27	1,181,437
–10+40 Mesh Feed				
BR-76-33	12,999	0.98	0.91	850,135
BR-76-34	12,793	0.94	0.78	804,680
BR-76-35	12,872	0.78	0.73	958,964
BR-76-36	12,902	0.65	0.60	833,469
Unscreened Feed				
BR-76-37	13,177	0.99	0.89	848,599
BR-76-38	12,976	0.99	--	948,546
BR-76-39	12,918	0.77	0.68	839,670
BR-76-40	12,903	0.68	0.43	824,502
–10+40 Mesh Feed + Lime				
BR-76-41	12,283	0.91	0.75	874,550
BR-76-42	11,918	0.69	0.49	657,159
BR-76-43	11,528	0.70	0.45	743,671
BR-76-44	11,297	0.55	0.28	659,971

BENCH-SCALE TEST WORK – CONCLUSIONS

The data obtained from the batch reactor confirmed the results of the thermobalance. Conclusions from this work included the following:

- *Satisfactory desulfurization of two Eastern U.S. coals was achieved.*
- *Terminal temperature of 1500°F and total reaction times of greater than 60 minutes are desirable.*
- *Rapid heat-up to terminal temperature, as in a fluidized-bed reactor, is satisfactory for sulfur removal.*
- *The use of the acceptor is not imperative, based on these tests.*
- *For satisfactory hydrodesulfurization, the coals should be pretreated.*
- *Hydrogen content of 50% or greater in the reacting gas is desirable; carbon monoxide is apparently equivalent to hydrogen in this regard.*
- *Hydrogen sulfide in the reacting gas tends to inhibit the hydrodesulfurization.*

10-INCH-DIAMETER CONTINUOUS FLUIDIZED-BED TEST WORK – HYDRODESULFURIZATION

RESULTS

The 10-inch fluidized-bed unit, used for continuous pretreatment tests, was utilized for hydrodesulfurization tests with Illinois No. 6 coal. Significant experimental difficulties precluded extensive data acquisition within the constraints of the program.

Data and conditions for two initial runs are shown in Table 39. Predefined bed temperatures and residence time were based on results from the batch reactor tests. The results for the run at 1450°F are suspect, because of operating problems – the unit had not previously been run at these high temperatures. A plug developed in the discharge line that would have caused some mixing of startup material and material from the steady-state period.

The second test, at 1300°F, shows the expected reduction of pyritic sulfur, but because the organic sulfur is not appreciably attacked, the total is high.

An inspection of the unit after these runs revealed that many of the heaters had burned out. These heaters must have failed during the high-temperature test and could have caused most of the operating problems. They were replaced and the unit prepared for additional runs.

A second supply of Illinois No. 6 coal was obtained; this coal was much higher in sulfur than the first sample.

A baseline run, using nitrogen as the fluidizing gas, was made at the same conditions, 1500°F and 2 hours residence time, specified for the first two hydrogen runs. Sulfur species of the pretreated coal and the nitrogen-treated coal are listed in Table 40. The data were not reduced because comparison of sulfur values, by type, was of interest for the test. The overall reduction of sulfur content was minimal, and organic sulfur in the treated coal is nearly the same as in the feedstock. Although the majority of the pyritic and sulfate types of sulfur have been reduced to sulfide, the total sulfur quantity of pyritic, sulfate, and sulfide types remains nearly constant. Treatment with nitrogen therefore does not remove sulfur significantly.

Table 41 lists the results of the three tests made in the 10-inch unit with hydrogen. The first two runs were made at 1500°F, and the last one at 1400°F. Residence time for all runs was 2 hours. Sulfur has not been adequately reduced in any of the tests. All tests show that sulfide-type sulfur has been produced, but not subsequently reduced. Neither has the organic sulfur been sufficiently removed. Material recovery is 68% to 70% of the original raw coal feed, with the fines from the cyclone calculated as product.

Some explanation of the reason for the poor sulfur removal may be seen in the gas analyses of Table 42. These show an average sulfur content in the gas near 4000 ppm or

TABLE 39. 10-INCH UNIT DATA – PRETREATED ILLINOIS NO. 6 COAL

Run No.	Feed Coal	Pretreated Coal	PDS-76-1	PDS-76-2
Temperature, °F		750	1450	1300
Residence Time, min		30	90	90
Sulfur, wt %				
Sulfide	0.01	0.01	0.89	0.45
Sulfate	0.13	0.04	0.00	0.07
Pyritic	0.83	0.64	0.06	0.08
Organic	1.43	1.50	1.12	0.98
Total	2.45	2.19	2.07	1.58
Weight Loss		9.6		
Reduced Data				
Weight, lb	100.0	90.4	54.2	57.2
Sulfur Weight, lb				
Sulfide	0.01	0.01	0.48	0.26
Sulfate	0.13	0.04	0.00	0.04
Pyritic	0.83	0.58	0.03	0.05
Organic	1.48	1.34	0.61	0.56
Total	2.45	1.97	1.12	0.91
Sulfur Content, %				
Sulfide	0.01	0.01		
Sulfate	0.13	0.04		
Pyritic	0.83	0.64	0.06	0.08
Organic	1.43	1.50	1.12	0.98
Total	2.45	2.19	1.18	1.06
Sulfur Removal, wt %				
From Feed			67.5	69.0
From Coal		19.6	73.9	75.1

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TABLE 40. SULFUR SPECIES IN NITROGEN RUN

Sulfur, wt %	Pretreated Coal	NDS-1
Sulfide	0.02	0.85
Sulfate	0.15	0.03
Pyritic	0.98	0.15
Organic	2.24	2.05
Total	3.39	3.08

TABLE 41. 10-INCH UNIT RUNS WITH PRETREATED ILLINOIS NO. 6 COAL

	Raw Coal	Pretreated Coal	PDS-77-1	PDS-77-2	PDS-77-3
Temperature	--	750	1500	1500	1400
Residence Time, min	--	30	120	120	120
Sulfur, wt %					
Sulfide	0.01	0.02	0.72	1.24	1.13
Sulfate	0.02	0.15	0.05	0.01	0.01
Pyritic	1.87	0.98	0.28	0.11	0.05
Organic	2.59	2.24	1.35	0.93	0.86
Total	4.49	3.39	2.40	2.29	2.05
Weight, lb					
Initial	100.00	--	90.1	90.1	90.1
Treated	--	90.1	69.6	67.9	70.2
Weight Loss, %					
Raw Coal	--	9.9	30.4	32.1	29.8
Pretreated Coal	--	--	22.7	24.6	22.1
Reduced Data					
Weight, lb	100.00	90.9	69.6	67.9	70.2
Sulfur Weight, lb					
Sulfide	0.01	0.02	0.50	0.84	0.79
Sulfate	0.02	0.14	0.03	0.01	0.01
Pyritic	1.87	0.89	0.19	0.07	0.04
Organic	2.59	2.04	0.94	0.63	0.60
Total	4.49	3.09	1.66	1.55	1.44
Sulfur Content, %					
Sulfide	0.01	0.02	--	--	--
Sulfate	0.02	0.15	--	--	--
Pyritic	1.87	0.98	0.28	0.11	0.05
Organic	2.59	2.24	1.35	0.93	0.86
Total	4.49	3.39	1.63	1.04	0.91
Sulfur Removal, wt %					
From Pretreated Coal	--	--	63.4	77.3	79.2
From Raw Coal	--	24.5	74.8	84.4	85.7

TABLE 42. GAS ANALYSIS

Mass Spectrograph Analysis

	PDS-77-1	PDS-77-2		PDS-77-3	
	Off-Gas	Feed Gas	Off-Gas	Feed Gas	Off-Gas
	mol %				
CO	--	2.14	4.70	0.12	4.76
CO_2	--	0.04	0.40	0.03	4.63
H_2	--	97.14	92.01	99.85	91.22
Argon	--	0.09	0.11	--	0.32
Methane	2.32	0.59	2.68	--	3.17
Ethane	0.02	--	0.04	--	0.07
Others C_3+	--	--	0.06	--	--

Sulfur Analysis

ppm as	PDS-77-1	PDS-77-2	PDS-77-3
H_2S	3167	5533	4347
CS_2	194	--	--
Thiophene	543	--	--

more in each case. As was proved concurrently in the batch reactor tests, sulfur removal is reduced when the back pressure of H_2S is significant.

Another major factor may be the amount of hydrogen used. In the batch reactor tests that showed good sulfur reduction, hydrogen usage was 240 SCF/lb of coal. However, in the 10-inch unit, with the hydrogen rate specified by mechanical and fluidization characteristics, the relative hydrogen usage was only 78 SCF/lb of coal.

The two possible causes of poorer removal – lower H_2/coal ratio and higher H_2S back pressure – are interrelated. Discharge of the same amount of H_2S into less hydrogen will increase its H_2S back pressure. Note that with increased scale-up, the relative hydrogen/coal ratio falls still further: The hydrogen rate is determined by the bed cross-sectional area, but the coal rate is determined by the bed volume.

Earlier in the program, the decision had been made to eliminate the acceptor from the process. Thermobalance and batch reactor tests had indicated that the acceptor was unnecessary and that significant process simplification could be realized by not using the acceptor. However, the scale-up problem, discussed above, now indicates that the acceptor may be required in the process.

Sulfur balances for the runs are shown in Table 43. These results show good detection of sulfur by the on-line chromatograph. Much better detection of H_2S is experienced than when grab gas samples were taken to be analyzed later. Differences in the sulfur balances (+2% to -10%) are reasonable, considering the difficulty of sampling and analysis of reduced sulfur compounds, and the physical characteristics of the system.

CONTINUOUS RUNS – CONCLUSION

The data from the 10-inch-diameter, continuous-feed reactor are insufficient to refute or to support the conclusion from the laboratory- and bench-scale test work. One fact, however, is apparent — the sulfur-getter concept is required in the system. The high back-pressure of H_2S in larger scale equipment inhibits the desulfurization of the coal.

TABLE 43. SULFUR BALANCE FOR PDS RUNS

Run No.	PDS-77-1	PDS-77-2	PDS-77-3
Sulfur In, lb			
Coal Feed (12.37 lb/hr)	0.42	0.42	0.42
Total In	0.42	0.42	0.42
Sulfur Out, lb			
Product	0.23	0.21	0.20
Gases	0.17	0.22	0.18
Total Out	0.40	0.43	0.38

NITROGEN EMISSIONS FOR TREATED COALS

Coal combustion tends to convert the nitrogen content in the coal to NO_x. Additional NO_x is formed from atmospheric nitrogen at combustion temperatures. The present Federal Standard is 0.7 lb of NO_x total, from both sources, per million Btu. Reductions of the nitrogen content of the fuel would also reduce the NO_x emissions during combustion. Some of the data from batch reactor runs were examined to determine if significant nitrogen removal had been accomplished. Table 44 and Figure 18 present the results. The nitrogen content of the product fuel is about one-half of the content of the raw coal, or, considering the treated material recovery, approximately three-quarters of the nitrogen has been removed from the raw coal. The nitrogen emissions are shown in Table 44 for the tests on Western Kentucky No. 9 coal. The operating conditions for these runs are given in Table 45. On a 1 million Btu basis, the amount of nitrogen has been reduced from 1.23 lb to about 0.50 to 0.60 lb. Figure 18 shows the percent of nitrogen remaining per million Btu and the effect of the terminal temperature.

TABLE 44. NITROGEN EMISSIONS FOR BATCH REACTOR TESTS
WESTERN KENTUCKY NO. 9 COAL

	% N_2	Btu/lb	$N_2/10^6$ Btu
Coal (Dry)	1.53	12,454	1.23
Pretreated +40 Mesh	1.48	11,809	1.25
BR-76-1, Residue	1.13	12,119	0.93
BR-76-2, +50 Mesh	0.95	12,107	0.78
Float	0.93	12,684	0.73
BR-76-3, Residue	0.78	11,967	0.65
BR-76-4, +50 Mesh	0.61	11,035	0.55
Float	0.74	12,134	0.61
BR-76-5, Residue	0.65	11,651	0.56
BR-76-6, +50 Mesh	0.59	11,463	0.51
Float	0.61	12,029	0.51
BR-76-7, Residue	0.73	11,565	0.63
BR-76-8, +50 Mesh	0.56	10,530	0.53
Float	0.62	11,789	0.53

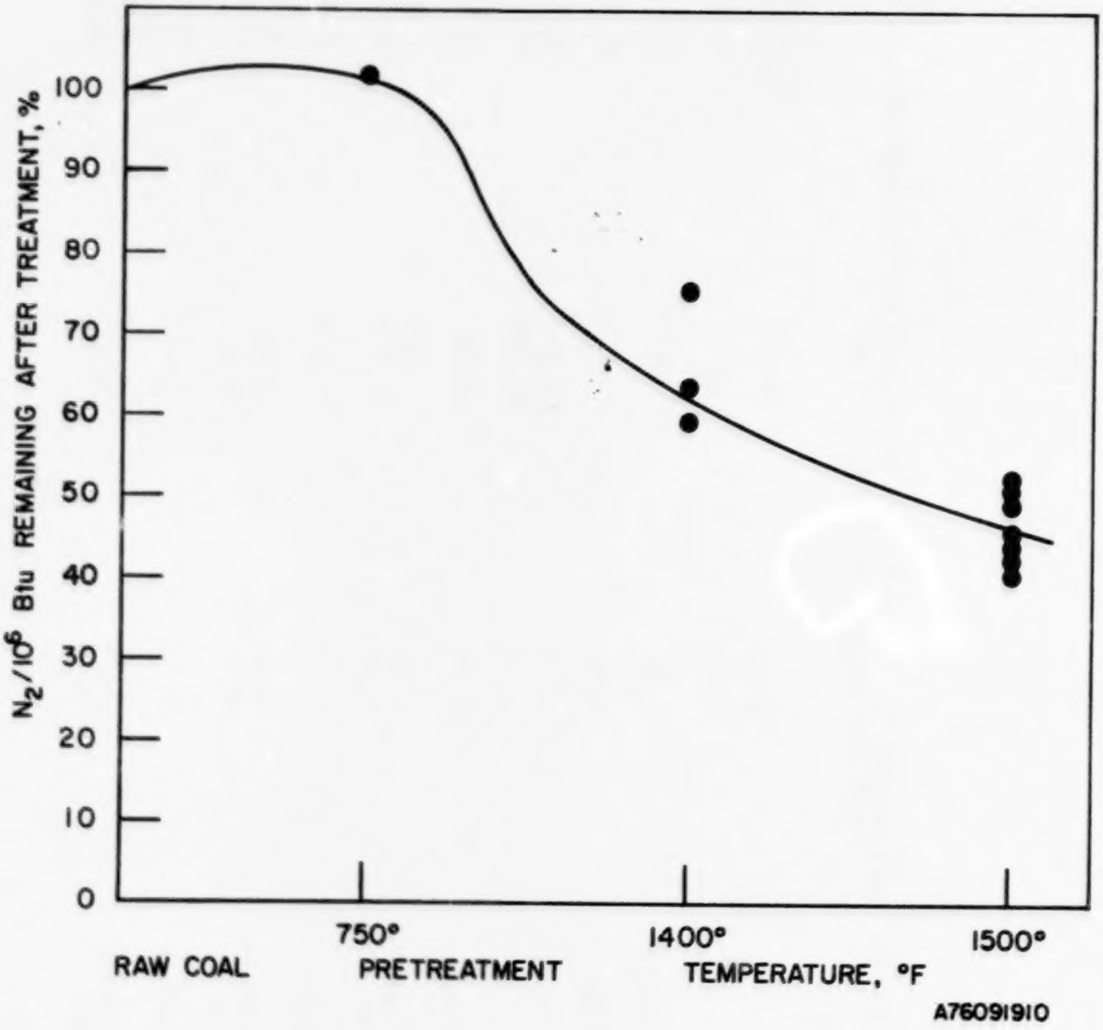

Figure 18. Percent of nitrogen remaining after treatment, for batch reactor tests with Western Kentucky No. 9 coal.

TABLE 45. BATCH REACTOR TEST RUN DATA FOR PRETREATED WESTERN KENTUCKY NO. 9 COAL

Run No.	Feed Coal*	+40 Mesh Pretreated Coal	BR-76-1		BR-76-2				
Lime/Coal Feed Ratio			0:1		2:1				
Coal	W. Ky. No. 9		Pretreated W. Ky. No. 9						
Heating Rate, °F/min			5		5				
Terminal Temperature, °F		750	1400		1400				
Holding Time, min		30	30		30				
			Feed	Residue	Feed	+50	−50	Float	Sink
Sulfur, wt %									
Sulfide	0.02	0.04	0.04	0.42	0.01	0.11	1.01	0.05	1.02
Sulfate	0.60	0.10	0.10	0.00	0.03	0.01	0.04	0.01	0.04
Pyritic	1.06	1.54	1.54	0.02	0.52	0.02	0.00	0.01	0.00
Organic	1.82	1.38	1.38	0.81	0.46	0.30	0.06	0.06	0.21
Total	3.50	3.06	3.06	1.25	1.02	0.44	1.11	0.13	1.27
Weight, g									
Initial	100.00		150		150				
Treated		90.84		109.8		126.4			
Weight Loss, %									
Total Weight		9.16		26.8		15.73			
Coal Weight		9.16		26.8		47.20			
Reduced Data									
Weight, lb			90.84	66.49	272.52	61.39	168.26	57.97	171.68
Sulfur Weight, lb									
Sulfide	0.02	0.04	0.04	0.30	0.04	0.07	1.70	0.03	1.75
Sulfate	0.60	0.09	0.09	0.00	0.09	0.01	0.07	0.01	0.07
Pyritic	1.06	1.40	1.40	0.01	1.40	0.01	0.00	0.01	0.00
Organic	1.82	1.25	1.25	0.54	1.25	0.18	0.10	0.03	0.36
Total	3.50	2.78	2.78	0.85	2.78	0.27	1.87	0.08	2.18
Sulfur Content %									
Sulfide	0.02	0.04	0.04		0.01				
Sulfate	0.60	0.10	0.10		0.03				
Pyritic	1.06	1.54	1.54	0.02	0.52	0.02		0.01	
Organic	1.82	1.38	1.38	0.81	0.46	0.30		0.06	
Total	3.50	3.06	3.06	0.83	1.02	0.32		0.07	
Sulfur Removal, wt %									
From Feed				80.2		93.2		98.6	
From Coal		20.6		84.3		94.6		98.9	

*Calculated for 40 mesh fraction.

TABLE 45. BATCH REACTOR TEST RUN DATA FOR PRETREATED WESTERN KENTUCKY NO. 9 COAL (Continued)

Run No.	Feed Coal*	+40 Mesh Pretreated Coal	BR-76-3		BR-76-4				
			Feed	Residue	Feed	+50	−50	Float	Sink
Lime/Coal Feed Ratio			0:1		2:1				
Coal	W. Ky. No. 9		Pretreated W. Ky. No. 9						
Heating Rate, °F/min			5		5				
Terminal Temperature, °F		750	1500		1500				
Holding Time, min		30	30		30				
Sulfur, wt %									
Sulfide	0.02	0.04	0.04	0.12	0.01	0.22	1.29	0.10	1.30
Sulfate	0.60	0.10	0.10	0.00	0.03	0.01	0.05	0.00	0.03
Pyritic	1.06	1.54	1.54	0.02	0.52	0.02	0.00	0.01	0.00
Organic	1.82	1.38	1.38	0.37	0.46	0.17	0.04	0.32	0.08
Total	3.50	3.06	3.06	0.51	1.02	0.42	1.38	0.43	1.41
Weight, g									
Initial	100.00		75.0		150				
Treated		90.84		51.7		121.9			
Weight Loss, %									
Total Weight		9.16		31.1		18.7			
Coal Weight		9.16		31.1		56.2			
Reduced Data									
Weight, lb			90.84	62.62	272.52	53.17	168.29	49.68	171.78
Sulfur Weight, lb									
Sulfide	0.02	0.04	0.04	0.08	0.04	0.12	2.17	0.05	2.23
Sulfate	0.60	0.09	0.09	0.00	0.09	0.01	0.08	0.00	0.05
Pyritic	1.06	1.40	1.40	0.01	1.40	0.01	0.00	0.01	0.00
Organic	1.82	1.25	1.25	0.23	1.25	0.09	0.07	0.16	0.14
Total	3.50	2.78	2.78	0.32	2.78	0.23	2.32	0.22	2.42
Sulfur Content %									
Sulfide	0.02	0.04	0.04		0.01				
Sulfate	0.60	0.10	0.10		0.03				
Pyritic	1.06	1.54	1.54	0.02	0.52	0.02		0.01	
Organic	1.82	1.38	1.38	0.37	0.46	0.17		0.32	
Total	3.50	3.06	3.06	0.39	1.02	0.19		0.33	
Sulfur Removal, wt %									
From Feed				91.4		96.4		93.9	
From Coal		20.6		93.1		97.1		95.1	

*Calculated for 40 mesh fraction.

TABLE 45. BATCH REACTOR TEST RUN DATA FOR PRETREATED WESTERN KENTUCKY NO. 9 COAL (Continued)

Run No.	Feed Coal*	+40 Mesh Pretreated Coal	BR-76-5		BR-76-6				
Lime/Coal Feed Ratio			0:1		2:1				
Coal	W. Ky. No. 9		Pretreated W. Ky. No. 9						
Heating Rate, °F/min			20		20				
Terminal Temperature, °F		750	1500		1500				
Holding Time, min		30	240		240				
			Feed	Residue	Feed	+50	−50	Float	Sink
Sulfur, wt %									
Sulfide	0.02	0.04	0.04	0.26	0.01	0.13	1.02	0.06	0.95
Sulfate	0.60	0.10	0.10	0.01	0.03	0.00	0.01	0.01	0.03
Pyritic	1.06	1.54	1.54	0.02	0.52	0.00	0.00	0.03	0.00
Organic	1.82	1.38	1.38	0.27	0.46	0.09	0.02	0.19	0.02
Total	3.50	3.06	3.06	0.56	1.02	0.22	1.05	0.29	1.00
Weight, g									
Initial	100.00		75		150				
Treated		90.84		41.3		119.4			
Weight Loss, %									
Total Weight		9.16		44.93		20.4			
Coal Weight		9.16		44.93		61.2			
Reduced Data									
Weight, lb			90.84	50.02	272.52	53.96	162.97	48.01	168.92
Sulfur Weight, lb									
Sulfide	0.02	0.04	0.04	0.13	0.04	0.07	1.66	0.03	1.60
Sulfate	0.60	0.09	0.09	0.01	0.09	0.00	0.02	0.00	0.05
Pyritic	1.06	1.40	1.40	0.01	1.40	0.00	0.00	0.01	0.00
Organic	1.82	1.25	1.25	0.14	1.25	0.05	0.03	0.09	0.03
Total	3.50	2.78	2.78	0.29	2.78	0.12	1.71	0.13	1.68
Sulfur Content %									
Sulfide	0.02	0.04	0.04	--	0.01	--		--	
Sulfate	0.60	0.10	0.10	--	0.03	--		--	
Pyritic	1.06	1.54	1.54	0.02	0.52	0.00		0.03	
Organic	1.82	1.38	1.38	0.27	0.46	0.09		0.19	
Total	3.50	3.06	3.06	0.29	1.02	0.09		0.22	
Sulfur Removal, wt %									
From Feed				94.6		98.2		96.4	
From Coal		20.6		95.7		98.6		97.1	

*Calculated for 40 mesh fraction.

TABLE 45. BATCH REACTOR TEST RUN DATA FOR PRETREATED WESTERN KENTUCKY NO. 9 COAL (Continued)

Run No.	Feed Coal*	+40 Mesh Pretreated Coal	BR-76-7		BR-76-8				
Lime/Coal Feed Ratio			0:1		2:1				
Coal	W. Ky. No. 9		Pretreated W. Ky. No. 9						
Heating Rate, °F/min			Rapid		Rapid				
Terminal Temperature, °F		750	1500		1500				
Holding Time, min		30	330		330				
			Feed	Residue	Feed	+50	−50	Float	Sink
Sulfur, wt %									
Sulfide	0.02	0.04	0.04	0.22	0.01	0.13	1.26	0.12	1.35
Sulfate	0.60	0.10	0.10	0.00	0.03	0.01	0.01	0.02	0.03
Pyritic	1.06	1.54	1.54	0.02	0.52	0.00	0.00	0.02	0.00
Organic	1.82	1.38	1.38	0.14	0.46	0.12	0.03	0.14	0.03
Total	3.50	3.06	3.06	0.38	1.02	0.26	1.30	0.30	1.41
Weight, g									
Initial	100.00		75		150				
Treated		90.84		51.2		108.4			
Weight Loss, %									
Total Weight		9.16		31.7		27.7			
Coal Weight		9.16		31.7		83.1			
Reduced Data									
Weight, lb			90.84	62.01	272.52	40.81	156.13	35.71	161.23
Sulfur Weight, lb									
Sulfide	0.02	0.04	0.04	0.14	0.04	0.05	1.96	0.04	2.18
Sulfate	0.60	0.09	0.09	0.00	0.09	0.00	0.02	0.01	0.05
Pyritic	1.06	1.40	1.40	0.01	1.40	0.00	0.00	0.01	0.00
Organic	1.82	1.25	1.25	0.09	1.25	0.05	0.05	0.05	0.05
Total	3.50	2.78	2.78	0.24	2.78	0.10	2.03	0.11	2.28
Sulfur Content %									
Sulfide	0.02	0.04	0.04		0.01	--			
Sulfate	0.60	0.10	0.10		0.03	--			
Pyritic	1.06	1.54	1.54	0.02	0.52	0.00		0.02	
Organic	1.82	1.38	1.38	0.14	0.46	0.12		0.14	
Total	3.50	3.06	3.06	0.16	1.02	0.12		0.16	
Sulfur Removal, wt %									
From Feed				96.4		98.2		97.8	
From Coal		20.6		97.1		98.6		98.3	

*Calculated for 40 mesh fraction.

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COAL COMBUSTION METHODS

The following discussion is presented to indicate the applicability of the treated, low-sulfur solid fuel in various types of combustion equipment. Several methods exist for burning solid fuels. These methods are divided into two basic groups: fuel bed and suspension burning.

The term "fuel bed" aptly describes the technique. A bed of burning fuel is supported by a grate, screen, or similar device, depending upon the circumstances. Part, and sometimes all, of the air for combustion flows through the grate to provide cooling to the metal parts. Fuel replenishment is normally provided by mechanical means. Ash or residue, in larger units, is also discharged either by mechanical movement or by the physical arrangement of the bed.

In an underfeed, single-retort type of burner, a mechanical cam or similar apparatus pushes fuel toward the end of the stoker and upward into the fuel bed. The bed is sloped away from this point, and the burning coal moves away from the feed point by gravity so that ash and residues are dumped at the side. Air flows upward through the bed. This kind of stoker has low capacity and will handle most bituminous coals and anthracite. Preferable coal sizing is from 3/4 inch to 2 inches, with no more than 50% at less than 1/4 inch.

A multiple-retort stoker has several lanes, or retorts, formed by pairs of sloping grates or tuyere stacks. Fuel is forced into the lanes, as with the single retort, and air is admitted along the tops of the grates. In some cases, the grates are given slight motion to agitate the bed for better burning and for residue dumping. This kind of stoker is best suited to caking coals with relatively high ash-softening temperatures. Coal sizing is up to 2 inches, with 30% to 50% through a 1/4-inch screen.

Grate stokers can be oscillating, dumping, moving, or stationary. They are of the cross-feed or overfeed type. In the cross-feed type, fuel is fed by gravity onto the grate and into the furnace. In the overfeed, or spreader type, the fuel is propelled into the furnace, and 30% to 50% of it is consumed while suspended above the bed. The remaining fuel falls to the bed to be burned. A spreader-feed-type stoker has the disadvantage that sometimes the ash can have a carbon content of 30% to 50% and must be recycled to achieve better fuel efficiency.

In all the grate stokers, air flows upward through the grate, to provide cooling, and into the feed bed. Additional air may be provided above the bed. Dumping of ash and residue depends on the type of grate. A stationary grate must be divided into zones for the burning and ash removal. The grates themselves can be dumped in a dumping-grate stoker, which eliminates the mechanical or hand holing of ashes. With travelling or oscillating grates, the bed is mechanically moved to the end opposite the feed, where the residue is dumped. A greater heat release, because of less ash on the grates, has made these two mechanical stokers more economical. Grate stokers are widely used because they can burn all types of coal from anthracite to lower rank bituminous coal and lignite.

Considering the characteristics and requirements of fuel beds, they would not be recommended for the proposed low-sulfur fuel unless the fine, desulfurized solids were first briquetted. Fuel capacity would rule out the single-retort stoker, and coal sizing would do the same for both of the retort-type stokers. Their fuel-particle-size requirements are much larger than the proposed low-sulfur fuel as produced directly. Also, if lime is used and not separated from the fuel before burning, the feeding and residue-removing systems must be larger to handle the increased ash load. The grate stokers could possibly use this fuel, but, once again, the particle size must be increased if lime is used and not separated. Another drawback is that because most of the air flows upward through the grate, some fluidization may occur in our small-sized fuel (unless briquetted). Overall, stoker-type fuel beds are not readily adaptable to this fuel and would probably require special design considerations.

Suspension burning means that pulverized fuel is burned while suspended in air or gas (unsupported mechanically). This type of combustion is used more extensively in the United States than the coarse-coal fuel beds. A greater steam-generating capacity is afforded by this method. Suspension burning is independent of coal-caking characteristics, and load changes can be made quickly. Several burner arrangements and firing methods are possible.

A fantail, vertical-firing method has a U-shaped flame. In this type of burner the fuel and primary air (used for coal transport) are forced downward into a vertical burning chamber. Secondary air for combustion is introduced at the sides of the chamber and shapes the U. Low-volatile chars or fuels with less than 15% volatiles are especially suited for this type of combustion because the fuel stream is projected well into the furnace before the introduction of the bulk of the combustion air.

The cyclone firing method uses a coarser coal, usually −4 mesh. The primary air and coal are admitted into the cylindrical chamber tangentially, but independent of the secondary air. Finer particles are burned in suspension, while the larger ones are thrown to the wall. A sticky coating of molten slag catches these particles and retains them until they are burned. Slag is continuously drained and quenched.

In other types of burners, the primary air used to transport the coal is about 20% of the total required. The secondary air is mixed with the coal and primary air in the burner and then enters the furnace. Burner arrangement usually depends on the requirements of the system. These types are normally associated with more highly volatile fuels with higher volatile counts.

In the last few years, another, more sophisticated type of suspension burning has been put into use – fluidized-bed combustion. The fluidized-bed combustor is a vertical vessel of appropriate size. Air enters a chamber in the bottom and flows upward through a distribution plate. The air-flow rate is controlled so that the fuel is fluidized and burned while suspended. Coal is introduced into the bed above the distribution plate, and residue is discharged continuously. The location of the feed and discharge points depends on the fuel, bed size, etc. Heat is transferred to an absorption surface, usually steam tubes that are located within the fluidized bed. Internal cyclones are used for removing dust from exit gases, and the fines are returned to the bed. Fluidized beds have several advantages. They run at lower temperatures, which results in less formation of nitrogen oxides and in the retention of some sulfur in the residue of some types of coal. Higher heat-transfer rates are possible with the transfer surface immersed in the bed. A pressurized system would also operate more efficiently than an atmospheric one.

Some of the suspension systems lend themselves to the proposed low-sulfur fuel more readily than the stoker types. Because of our proposed small-particle size, the fuel-suspension is easier. The vertical fantail, cyclone, or fluidized bed should be able to handle the fuel without much change from regular coal burning if the lime is separated before combustion. If the lime is left in for combustion, some size changes would be necessary, as well as an increase of fluidizing air. The units would be designed so that the lime could be collected and disposed of or recycled.

If it is decided to use lime in the process, two points must be considered to determine if the lime should be separated before burning. The first is whether it will be possible to maintain combustion and temperatures with the larger amount of inert material in the combustor; the second is the evaluation of the disposition and final form of the sulfur, captured by the lime during hydrogen treatment. At present, we do not know if that sulfur will be retained by the lime as $CaSO_4$, if it will be discharged as SO_2, or if the lime has sufficient capability to capture additional sulfur during combustion. Both of these questions will require investigation; however, they become academic if it is decided not to use lime in the hydrogen-treatment step of the process.

ECONOMIC STUDIES

A conceptual process design has been prepared. Material and heat balances were calculated for a 20,000-ton/day facility (Figure 19) and the plant cost was estimated by conventional engineering-factoring techniques. The process design basis was the system without the sulfur acceptor present, as defined from small-scale tests. Concurrent with this economic study, the large-scale experimental program determined that the acceptor was indeed required and the effects upon the overall process were approximated.

The design basis for the pretreater section of the facility was taken largely from this work, with definition of the off-gas composition and heat release determined from other, parallel programs at the contractor's pilot plant. The hydrodesulfurization temperature, residence time, sulfur removal, and carbon conversion were developed from the data reported here; the off-gas composition, benzene production, and energy requirements were developed from the contractor's in-house data base on hydropyrolysis and hydrogasification.

PROCESS DESIGN RESULTS

Run-of-mine coal (Western Kentucky No. 9) is fed at a rate of 20,000 tons/day. The coal is continuously stored, reclaimed from storage, and crushed to −14 mesh. The coal is then pretreated in a fluidized-bed reactor at 750°F, consuming 1 SCF O_2/lb coal. In this unit, 242.4 million Btu/hr is recovered to generate 1200 psig steam. The pretreater off-gas, with a higher heating value of over 100 Btu/SCF, has a flame temperature of about 2600°F (partially because of the 750°F initial temperature). This gas is partially used as process fuel where direct-fired heating is required, and the remainder is used to fire a power plant boiler. The power plant generates 155,000 kW, of which 95,700 kW is exported (as by-product). Boiler fuel gas is scrubbed to recover SO_2, which is sent to a sulfuric acid plant. The plant requires 3835 gpm of raw water for cooling tower and boiler feedwater makeup.

The pretreated coal is sent to a fluidized-bed reaction vessel (the hydrodesulfurization reactor) where it is desulfurized in a 50% hydrogen atmosphere. The reactions are endothermic and require 406.4 million Btu/hr of heat addition. The treated solid fuel product leaves the reaction vessel at 1500°F and is cooled in a rotary-kiln-type device where 342.5 million Btu/hr of heat is recovered.

The reactor off-gas is cooled to 200°F in a waste-heat recovery unit (recovering 406.9 million Btu/hr). The gas is then scrubbed in a venturi-type scrubber for dust removal. After cooling to 100°F and removal of condensed water, the gas is compressed to 35 psig for acid-gas removal. The acid-gas is sent to an H_2S burner where this SO_2 product is combined with the SO_2 from the pretreater off-gas and converted to 1928 tons/day of H_2SO_4 (100%). The sweet gas from the acid-gas removal unit is split into co-product gas (66 billion Btu/day) and gas required for recycle to the fluidized-bed reactor. The hydrogen content of this recycle gas is raised from 39% to 50% by steam reforming a slipstream of the recycle gas.

The recovery of the energy in the feed coal is as follows:

Solid Fuel Product	61.3%
Co-Product Fuel Gas (570 Btu/SCF)	14.0%
Light Oil By-Product	7.0%
Exported Power	1.7%
Recovered Energy	84.0%

Additionally, the facility will recover about 150 to 300 tons daily of anhydrous ammonia with additional heating value. (However, its major market will not be for fuel purposes.)

PROCESS ECONOMICS

Capital and operating costs are shown in Tables 46 and 47. The price of treated product is calculated using utility type financing at \$30/ton, or about \$1.25/million Btu. The basis for this calculation is shown in Table 48. Total capital required for the 20,000-ton/day plant is \$437 million. In order to make the process economically feasible, considerable capital has to be spent on equipment to recover by-products and convert the pretreater off-gas into power. This amounts to about \$90 million (or about 40% of the installed plant cost) including capital spent for power generation by burning pretreater off-gas at about \$50 million.

The project contingency has been increased to 30% because of the following factors. The internals of the hydrodesulfurization reactor's materials of construction is an area of uncertainty because of the severe environment: a minimum temperature of 2600°F, with sulfur-containing combustor gas inside the tubes and a 1500°F fluidized bed, fluidized by a 50% H_2 gas on the outside of the tubes. In addition, both ammonia and COS may be produced and require more downstream processing. Other hydrodesulfurization off-gas composition uncertainities also affect the project contingency. The normal contingency used is 15%; in this case 15% process development contingency has been added to this. This is about double the hydrodesulfurization reactor cost and increases total capital required by \$47 million.

Operating Costs

Coal is priced at \$1.00/million Btu and is the major operating cost. Gross operating costs are \$185 million per year. This is largely offset by the significant amount of by-product credit, \$119 million, for a net operating cost of \$66 million per year.

By-product credit unit costs are —

Power: 2.5¢/kWhr
H_2SO_4: \$34/ton of 100% acid
Medium-Btu gas: \$2.30/million Btu*
Benzene: 35¢/gal.

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*Escalated from \$2.26/million Btu to \$2.30 because of time difference. Data source: "Economics of Current and Advanced Gasification Process for Fuel Gas Product," EPRI AF-244, Project 239 Final Report, July 1976.

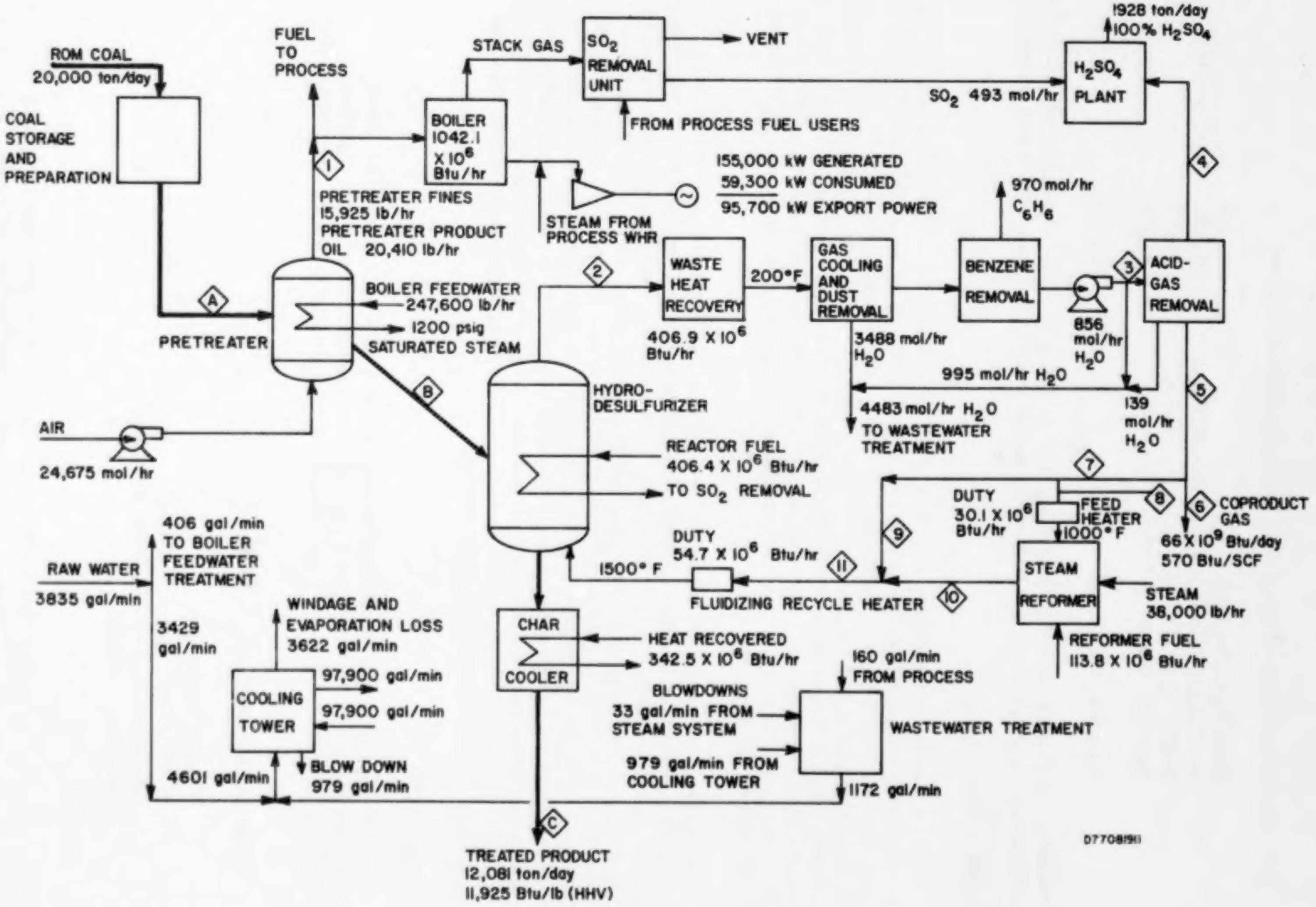
ROM COAL
20,000 ton/day
COAL STORAGE AND PREPARATION
A
PRETREATER
AIR
24,675 mol/hr
FUEL TO PROCESS
1
PRETREATER FINES 15,925 lb/hr
PRETREATER PRODUCT OIL 20,410 lb/hr
BOILER 1042.1 X 10^6 Btu/hr
STACK GAS
SO_2 REMOVAL UNIT
VENT
FROM PROCESS FUEL USERS
STEAM FROM PROCESS WHR
155,000 kW GENERATED
59,300 kW CONSUMED
95,700 kW EXPORT POWER
BOILER FEEDWATER 247,600 lb/hr
1200 psig SATURATED STEAM
B
2
HYDRO-DESULFURIZER
REACTOR FUEL 406.4 X 10^6 Btu/hr
TO SO_2 REMOVAL
WASTE HEAT RECOVERY
406.9 X 10^6 Btu/hr
200°F
GAS COOLING AND DUST REMOVAL
3488 mol/hr H_2O
4483 mol/hr H_2O TO WASTEWATER TREATMENT
BENZENE REMOVAL
970 mol/hr C_6H_6
856 mol/hr H_2O
995 mol/hr H_2O
3
ACID-GAS REMOVAL
139 mol/hr H_2O
SO_2 493 mol/hr
H_2SO_4 PLANT
1928 ton/day 100% H_2SO_4
4
5
6
COPRODUCT GAS
66 X 10^9 Btu/day
570 Btu/SCF
7
8
9
10
11
DUTY 30.1 X 10^6 Btu/hr
FEED HEATER
1000° F
STEAM REFORMER
STEAM 38,000 lb/hr
REFORMER FUEL 113.8 X 10^6 Btu/hr
DUTY 54.7 X 10^6 Btu/hr
1500° F
FLUIDIZING RECYCLE HEATER
CHAR COOLER
HEAT RECOVERED 342.5 X 10^6 Btu/hr
C
TREATED PRODUCT
12,081 ton/day
11,925 Btu/lb (HHV)
RAW WATER
3835 gal/min
406 gal/min TO BOILER FEEDWATER TREATMENT
3429 gal/min
4601 gal/min
WINDAGE AND EVAPORATION LOSS 3622 gal/min
COOLING TOWER
97,900 gal/min
97,900 gal/min
BLOW DOWN 979 gal/min
BLOWDOWNS
33 gal/min FROM STEAM SYSTEM
979 gal/min FROM COOLING TOWER
160 gal/min FROM PROCESS
WASTEWATER TREATMENT
1172 gal/min
D7708I911

Figure 19. Coal desulfurization process (Western Kentucky coal, feed rate 20,000 tons/day).

Stream Description	Pretreater Off-Gas	Hydro-Treater Off-Gas	Off-Gas to Acid-Gas Removal	Feed to Sulfuric Acid Plant	Sweet Gas	CO Product Gas	Recycle	Reformer Feed	Reformer By-Pass	Reformer Product	Fluid-izing Recycle
Stream Number	1	2	3	4	5	6	7	8	9	10	11
Temperature, °F	750	1500	100	100	115	115	115	115	115	1500	820
Pressure, psia	20	20	50	25	45	43	43	43	43	25	25
Component, mol/hr											
CO	888	2716	2716	--	2716	1765	951	287	664	787	1451
CO_2	3217	1677	1677	931	746	485	261	79	182	380	562
H_2	137	7618	7618	--	7618	4949	2669	804	1865	3513	5378
H_2O	7569	4761	417	82	196	128	68	21	47	1161	1208
CH_4	915	7764	7764	--	7764	5044	2720	819	1901	17	1918
N_2	19696	591	591	--	591	384	207	62	145	62	207
H_2S	--	1153	1153	1153	--	--	--	--	--	--	--
C_2H_6	577	--	--	--	--	--	--	--	--	--	--
C_3H_8	384	--	--	--	--	--	--	--	--	--	--
C_4H_{10}	238	--	--	--	--	--	--	--	--	--	--
C_6H_6	--	970	--	--	--	--	--	--	--	--	--
O_2	778	--	--	--	--	--	--	--	--	--	--
SO_2	439	--	--	--	--	--	--	--	--	--	--
Total	34888	27250	21936	2166	19631	12755	6876	2072	4804	5920	10724

Stream Description	Pretreater Feed	Pretreated Coal	Treated Product
Stream Number	A	B	C
Component			
Composition, % (Dry)			
C	70.00	70.67	80.04
H	4.54	3.80	0.92
O	8.95	8.54	0.49
N	1.53	1.33	0.75
S	3.74	3.10	0.58
Ash	11.24	12.56	17.21
Total, lb/hr	1,666,667*	1,378,367	1,006,773

* Includes 96,667 lb/hr moisture.

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Figure 19. Coal desulfurization process (Western Kentucky coal, feed rate 20,000 tons/day). (Continued)

TABLE 46. CAPITAL INVENTMENT SUMMARY
MID-1975

		$\$10^6$	$\$10^6$
Coal Storage and Preparation			13.4
Pretreatment			8.9
Hydrodesulfurization			17.0
Char Cooler			9.3
Waste Heat Recovery			12.6
Gas Cooling and Dust Removal			4.0
Benzene Removal			14.6
Process Heaters			2.0
Reformer			4.7
Acid-Gas Removal and Compressors			11.1
Sulfuric Acid Plant			15.1
SO_2 Removal Unit			13.3
Boiler			31.8
Turbine Generator			18.8
Electric Power Distribution at $170/kW; 59,300 kW			10.1
Cooling Towers & Plant Makeup Water			3.3
Waste Water Treating			4.8
Particulate Emission Control			3.7
General Facilities			22.1
Installed Plant Cost, Excluding Contingencies			220.6
Contingency 15% + 15% Process Development Contingency = 30%			66.2
Total Bare Cost			286.8
Contractors Overhead and Profits (15%)			43.0
Total Plant Investment (TPI)			329.8
Interest During Construction (9% X 1.875 years X TPI)			55.6
Start-up Cost (5% of TPI)			16.5
Working Capital:	a) 60 days coal at $20/ton	28.2	
	b) 0.9% of I	3.0	
	c) 1/24 of Treated Product Gas at $\$1.00/10^6$ Btu	3.9	
		35.1	35.1
Total Capital Required			437.0

TABLE 47. ANNUAL OPERATING COSTS

		$\$10^6$/yr
Operating Cost Components		
Coal Cost		
20,000 tons/day, 328.5 days/yr, at $\$1.00/10^6$ Btu (\$23.49/ton)		154.329
Other Direct Materials		0.058
Catalyts and Chemicals		1.533
Purchased Utilities		
Raw Water 3835 gpm at \$0.45/1000 gal		0.816
Labor		
(a) Process Operating Labor 37 men/shift X \$7.20/hr X 8760 hr/yr		2.334
(b) Maintenance Labor 1.5% of TPI		4.947
(c) Supervision 15% of (a) + (b)		1.092
(d) Administration and General Overhead 60% of (a) + (b) + (c)		5.024
Supplies		
Operating [30% of (a)]		0.700
Maintenance 1.5% of TPI		4.947
Local Taxes and Insurance 2.7% of TPI		8.905
Total Gross Operating Cost		184.685
By-Product Credit		
Power: 95,700 kW X 328.5 X 24 hr X \$.025/kWr =	.862	
H_2SO_4: 1928 tons/day X 328.5 day/yr X \$34/ton =	21.534	
Medium-Btu/gas: 66 X 10^9 Btu/day X 328.5 days/yr X $\$2.30/10^6$ Btu =	49.886	
Benzene: 970 mol/hr X 24 hr/day X $\frac{78.11\ \text{lb/mol}}{7.40\ \text{lb/gal}}$ X 328.5 days/yr X \$0.35/gal =	28.289	
	118.551	(118.551)
Net Operating Cost		66.134

Product Price, Utility type financing

$$\frac{66.134 + 0.1198\ (437) + 0.0198\ (35.1)}{12{,}081\ \text{tons/day X } 328.5\ \text{days/yr}} = \frac{119.181 \text{ X } 10^6}{3{,}968{,}609\ \text{tons/yr}} = \$30.03/\text{ton}$$

(Utility-type financing)

$$\frac{\$119.181 \text{ X } 10^6}{94.653 \text{ X } 10^{12}\ \text{Btu/yr}} = \$1.26/10^6\ \text{Btu}$$

TABLE 48. GAS COST CALCULATION BY UTILITY METHOD USED IN THE "FINAL REPORT OF THE FPC SUPPLY-TECHNICAL ADVISORY TASK FORCE – SYNTHETIC GAS-COAL"

BASIS	UTILITY METHOD
Project Life	20 years
Depreciation	5%/year, straight line
Debt/Equity Ratio	75%/25%
Return on Equity	15%
Interest Rate on Debt	9%
Federal Income Tax	48%
Interest During Construction	Interest Rate (9%) X 1.875 years* X total plant investment
OTHER FACTORS	
Plant Stream Factor	90%
Contingencies	30% of installed plant cost (includes process development contingency of 15%)
Contractor's Overhead and Profits	15% of total bare costs
Start-up Cost	5% of total plant investment
Working Capital	a) Coal inventory (60 days feed at full rate) b) Material and supplies (0.9% of total plant investment) c) Net receivables at 1/24 X annual revenue required

Derived equation

$$\text{20-year average gas price, } \$/10^6 \text{ Btu} = \frac{N + 0.1198\ C + 0.0198\ W}{G}$$

Where

N = Net annual operating cost
C = Total capital required
W = Working capital
G = Annual treated product production

* 10% for 3 years, 90% for 1.75 years

The power credit is conservatively estimated, based on costs from a large, new power plant. The acid price is about 70% of market price. The price of the 570 Btu/SCF gas was set at $2.30/million Btu to be conservative. This is a price that was obtained from a study employing a modern fluidized-bed technology for producing low-Btu gas. In this study, coal was priced at $1.00/million Btu, and the time basis was mid-1975 for capital costs. The medium-Btu gas in this study probably could be sold for more than this. Benzene quality is unknown so 44% of value price was used.

Product Price Sensitivities

A change of $10 million in total capital required results in a treated product price change of 42¢/ton or 14¢/million Btu of treated product.

A change of $1.00/ton of raw coal cost results in a change of $1.72/ton in treated product price.

A change of $10 million in by-product credit or operating cost results in a $2.52/ton change in treated product price.

Effect of Process Revision to Include "Sulfur-Getter" Loop

Larger scale test results late in the program indicated that the initial sulfur-getter concept is still required (contrary to smaller scale tests that had indicated the elimination of this approach). The effect of a sulfur-getter loop (based on data from the contractor's Steam-Iron Process experience) can be conceptualized as follows.

The pretreated char is mixed with relatively unreactive iron ore in the hydrodesulfurization reactor. Unreactive ore is required to minimize the H_2-FeO reaction but still permit the H_2S-FeO reaction. Experience with the the contractor's Steam-Iron reaction system indicates that this approach is feasible.

The iron ore acts as an acceptor, reducing the back pressure of H_2S in the reactor and permitting the desulfurization reaction to approach the conditions experienced in the smaller scale tests (with lime or FeO acceptor).

The treated coal and iron ore mixture is cooled as in the base case above and the iron ore is separated from the coal magnetically. The ore is regenerated with steam (similar to the steam-iron reaction), producing an H_2S-rich gas that would be combined with the hydrodesulfurized hot raw product gas in the system of Figure 19. Any hydrogen that reacted with the iron ore is also recovered, perhaps eliminating the need for the reformer in the system. The H_2S would be recovered in the acid-gas removal section.

The additional equipment required for this acceptor loop includes a larger hydrodesulfurization (HDS) reactor, larger char cooler, magnetic iron separator, and iron ore regenerator. Operating costs would include steam lost to condensation, increased waste water treatment, and heat requirements for the regenerator.

For the purpose of an initial evaluation, the capital requirement of the facility is increased 25%, the by-product electrical power is eliminated, and 50% of the BTX is required for steam superheat feeding the iron ore regenerator. On this basis, the total capital requirement increases to $546 million and the process efficiency decreases to 78.8%. The cost of product increases to $1.75/million Btu. Even with the relatively

conservative estimated basis employed, the process produces a clean fuel that is significantly less costly than alternatives.

ECONOMIC STUDIES – CONCLUSION

Based on preliminary economic analysis, the commercial-scale process appears to have economic potential for the production of an environmentally-preferred solid fossil fuel. Product prices (mid-1975 basis) are anticipated to be about $1.25 to $1.75/million Btu.

PROCESS DEVELOPMENT UNIT (PDU) REQUIREMENTS

A conceptual design and initial equipment specification have been prepared for a Process Development Unit (PDU) for the coal desulfurization process. This unit is designed to incorporate the major features of the process and to operate on an integrated basis, at a 10 tons/day feed rate, to prove the technical feasibility of the system. The design is not detailed; additional data are required from the 10-inch-diameter unit before this detail can be developed. Rather, this initial design is presented to indicate the type of experimental equipment that would be necessary for such a system (as contrasted to the commercial plant concept discussed earlier). Figures 20 and 21 and Tables 49 and 50 present the process configuration.

CONCEPTUAL DESIGN

Coal, as received by railroad car or truck, is put into a storage bin to prevent weathering by the elements. Proper sizing is done by screening, grinding the oversize, and rescreening. The coal after screening goes to the feed bin for use in the process.

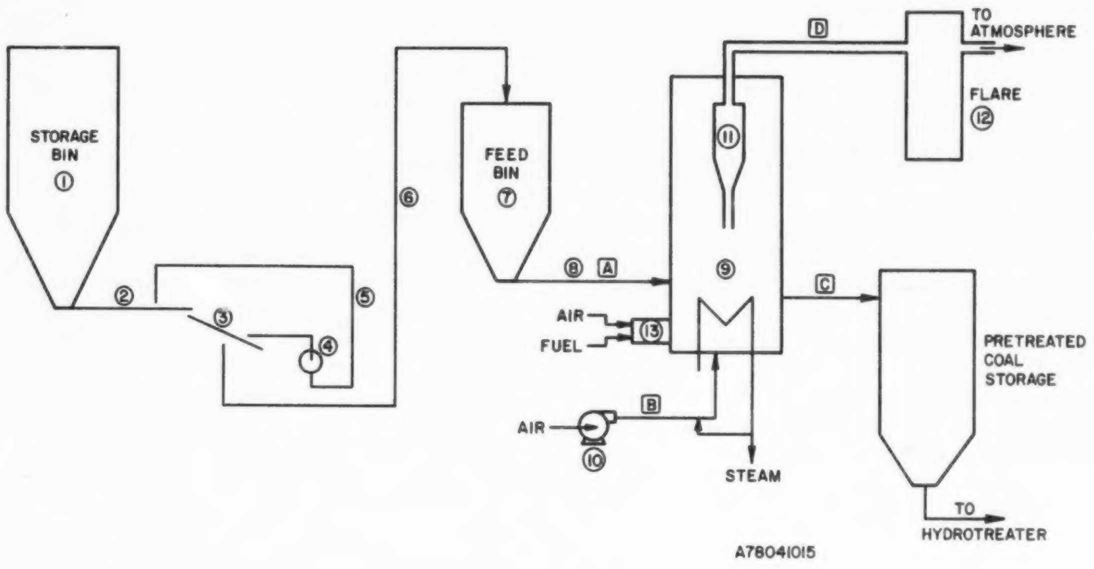
A variable-speed screw conveyor feeds the coal into the pretreater at the selected rate. The pretreater is equipped with a start-up heater, to reach the operating temperature, and a fan or compressor, to supply air for fluidization and reaction. Dust is removed from the off-gases by an internal cyclone that returns the fines to the bed; the vaporized tars, oils, and low-Btu gas are consumed by flaring. Pretreated coal is taken from the bed to storage before feeding to the hydrotreatment section.

In the hydrotreatment section the acceptor is stored in a feed bin for use in the system. Acceptor is fed either directly into the hydrotreater or it can be put into the pretreater coal feed system in the proper ratio of acceptor to coal. The hydrodesulfurizer is equipped with a heater to aid in start-up and to supply heat during operation if necessary. Hydrogen is also preheated before introduction to the hydrodesulfurizer. Solids are removed from the gases and returned to the bed by an internal cyclone.

The hydrotreater off-gases are cooled, scrubbed by di-glycol-amine (DGA) for sulfur and CO_2 removal, and split into two streams. One stream of the purified off-gas is sent to the incinerator and the other is recycled to the hydrotreater. Fresh H_2 is used as makeup to keep the H_2 pressure in the recycle gas at the proper value.

The char-acceptor mix is quenched and put through a magnetic separator. The acceptor is sent to a steam regenerator and provision is included for recycling to the feed bin. The treated solid fuel is discarded after sampling.

Table 51 lists the required capacities for the equipment in the pretreatment section. The raw coal storage bin is sized for 30 tons of coal, an equivalent of 3 days' run. The screening, milling, and recycle systems are sized so that feed material can be prepared in a relatively short time. Capacity for the pretreater feed bin is also 30 tons, enough for a 3-day run. The feed screw to the pretreater is capable of 0 to 2 tons/hr, which gives us the desired flexibility to adjust holding time in the system for various


TO ATMOSPHERE
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FLARE
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FEED BIN
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⑨
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C
②
③
⑤
④
AIR
FUEL
⑬
B
AIR
⑩
STEAM
PRETREATED COAL STORAGE
TO HYDROTREATER
A78041015

Figure 20. Pretreatment section.

TABLE 49. PRETREATER STREAMS

Stream	A	B	C	D
	lb/hr			
Ash	96.9		90.9	5.9
Carbon	590.5		543.9	13.8
Hydrogen	39.6		30.3	0.7
Sulfur	34.3		25.5	0.8
Nitrogen	12.9		12.2	0.3
Oxygen	75.8		69.4	3.6
Total	850.0		772.2	25.1
	mol/hr			
CO				0.450
O_2		2.24		0.054
CO_2				0.646
H_2				0.061
CH_4				0.231
C_2H_6				0.088
C_3H_8				0.044
C_4H_{10}				0.151
$>C_5$				
N_2		8.43		8.43

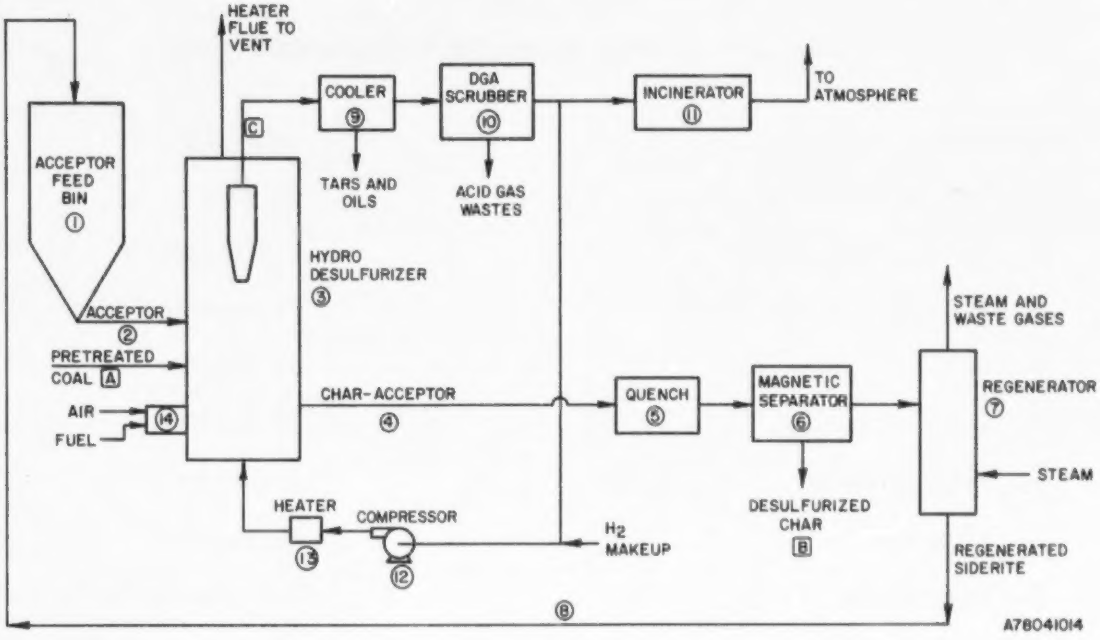
HEATER FLUE TO VENT
COOLER ⑨
DGA SCRUBBER ⑩
INCINERATOR ⑪
TO ATMOSPHERE
C
ACCEPTOR FEED BIN ①
TARS AND OILS
ACID GAS WASTES
HYDRO DESULFURIZER ③
STEAM AND WASTE GASES
ACCEPTOR ②
PRETREATED COAL A
AIR
FUEL
⑭
CHAR-ACCEPTOR ④
QUENCH ⑤
MAGNETIC SEPARATOR ⑥
REGENERATOR ⑦
STEAM
DESULFURIZED CHAR B
HEATER ⑬
COMPRESSOR ⑫
H_2 MAKEUP
REGENERATED SIDERITE
⑧
A78041014

Figure 21. Hydrotreatment section.

TABLE 50. HYDROTREATER STREAMS

Stream	A	B	C
		lb/hr	
Ash	90.9	90.9	
Carbon	543.9	437.3	
Hydrogen	30.3	6.9	
Sulfur	25.5	3.2	
Nitrogen	12.2	5.3	
Oxygen	69.4	8.9	
Total	772.2	552.5	
		mol/hr	
CO			15.07
CO_2			14.66
H_2			316.62
CH_4			1.01
C_2H_6			10.04
$>C_3$			0.22

TABLE 51. PRETREATER EQUIPMENT LIST

Equipment	Capacity
1. Raw Coal Storage	30 tons
2. Screen Feed Screw	0-5 tons/hr
3. Screener (single deck)	15 tons/hr
4. Mill	10 tons/hr
5. Recycle System	10 tons/hr
6. Coal Elevator	5 tons/hr
7. Feed Bin	30 tons/hr
8. Feed Screw	0-2 tons/hr
9. Pretreater	850 lb/hr
10. Air Compressor	10,000 SCF/hr at 5-10 psig
11. Cyclone	400 ACF/min at 750°F
12. Flare	400 ACF/min at 750°F (2110 Btu/SCF)
13. Start-up Heater	0.5 X 10^6 Btu/hr
14. Pretreater Coal Storage Bin	30 tons

coals. A residence time of 1 hour in the pretreater with 1 SCF O_2/lb of coal gives a bed requirement of 3.0-feet diameter by 4.5 feet height and an air flow of 4125 SCF/hr. The remainder of the fluidizing gas can be made up with steam. If a coal is used requiring more severe pretreatment, the bed height, and therefore residence time, must be increased. Provisions must be made, then, for an increase in bed depth, and also the height must be such that the cyclone can be internally mounted. The cyclone would be about 1 foot in diameter by about 4 feet tall. This indicates a pretreater 3 feet in diameter by at least 15 feet tall to accommodate a possible 9-foot tall bed and the cyclone with dipleg. The startup heater should have an input of 0.5 million Btu/hr. Off-gases would be consumed in a flare designed to also burn the intermediate-Btu by-product gas from the hydrotreater.

Pretreated coal would be fed to the hydrotreating system. Equipment lists and capacities for this section are shown in Table 52. Acceptor, if used, would be added either to the hydrotreater or into the pretreated coal feed system. An acceptor feed bin of 30 tons would supply 3 days at a 1:1 acceptor-to-coal weight ratio. The acceptor feed system should be capable of 0 to 2 tons/hr to cover a range of acceptor ratios. At a 1:1 ratio the hydrodesulfurizer must handle 1544 lb of mix per hour. A 3-hour residence time and fluidization velocities of up to 3 ft/s for the siderite requires a bed 7 feet in diameter and about 3.5 feet tall. The overall height of the vessel must be such that an internal cyclone can be included. To handle the hydrogen flow at the operating temperature the cyclone is 3.5 feet in diameter and 14 feet long. The overall hydrotreater should be 7 feet in diameter and 20 feet tall to accommodate the cyclone.

Off-gases are cooled by a cooler with a capacity of 4 million Btu/hr, then scrubbed with DGA. A slipstream of gas is pulled off, the amount determined by the gas analysis, to be flared. Makeup hydrogen is added to keep the partial pressure hydrogen in the hydrodesulfurizer feed gas at desired values. Solid material is quenched and then separated magnetically. Provision has been included for the acceptor to be regenerated by steam and recycled.

Heat for the process comes from the hydrogen heat at 4 million Btu/hr and an auxiliary process heater of 0.5 million Btu/hr.

This PDU will produce approximately 13,250 pounds of treated fuel, depending upon feed coal characteristics, from 10 tons of coal per day.

This unit is designed for experimental purposes – to prove the technical feasibility of the integrated system. It is not the fully integrated plant that was conceptualized for the commercial analysis. For example, low- and intermediate-Btu by-product gases are flared, rather than recovered for energy value; wastewater treatment is not included; and makeup hydrogen is used rather than treating a portion of the recycle gas for use. Rather, the PDU is designed for data acquisition on the key elements of the system.

TABLE 52. HYDROTREATER EQUIPMENT LIST

Equipment	Capacity
1. Acceptor Feed Bin	30 tons
2. Acceptor Feed Conveyor	0-2 tons/hr
3. Hydrodesulfurizer	1544 lb mix/hr
4. Char-Acceptor Discharge Conveyor	0-2 tons/hr
5. Solids Quench	0.75 X 10^6 Btu/hr
6. Magnetic Separator	1325 lb/hr
7. Regenerator	772 lb/hr
8. Regenerated Siderite Conveyor	772 lb/hr
9. Gas Cooler	4 X 10^6 Btu/hr
10. Scrubber-DGA for CO_2	2000 SCF/min
11. Flare	Use Pretreater Flare
12. Hydrogen Compressor	2000 SCF/min at 5-10 psig
13. Hydrogen Heater	4 X 10^6 Btu/hr
14. Process Heater	0.5 X 10^6 Btu/hr

FUTURE WORK

Assessment of the data now available, including the economic analysis, indicates that the process has merit for converting coal into a solid fossil fuel that can be directly consumed in accordance with present environmental regulations. The suggestions for future work include the following.

10-INCH-DIAMETER UNIT

The data from the continuous-feed, 10-inch-diameter reactor are insufficient to prove the process on that scale. Additional testing is required, with alternative sulfur-acceptor materials included in the system.

ACCEPTOR REGENERATION

Experimental data are required to prove the regeneration processing required for the preferred sulfur acceptor, or applied to this overall process.

PROCESS DEVELOPMENT UNIT

The Process Development Unit (PDU) described in this report, or a variation thereof as determined by the experimental program outlined above, should be constructed and operated to prove the technical feasibility of the process on an integrated, systematic basis.